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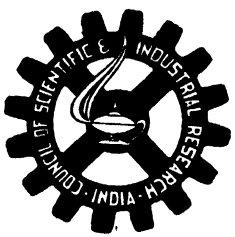

REGIONAL RESEARCH LABORATORY, HYDERABAD-9.

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INTRODUCTION

The Annual Report for 1958-59 follows in general the line of the one for 1957-58.

A significant development has been the increase in the number of problems undertaken on behalf of and sponsored by the industry, which is indicative of the growing interest by large, medium and small scale industries in the work of the Laboratory, and their keenness to take assistance in solution of their problems. A number of individuals and parties have also approached the Laboratory for assistance in setting up of new industries. This puts greater strain on the resources and talent available. Research workers, chemical engineers and auxiliary staff are called upon to establish working conditions, undertake design, installation and costing for industrial projects and render further assistance by way of procurement of equipment, installation and trial runs. The question as to what part of the programme of Laboratory should be devoted to ad hoc problems of the industry without adversely effecting the long-range programmes is important. A certain minimum effort is also essential for fundamental scientific work to keep up the tone of research in the Laboratory.

The number of pilot plants has increased further. Every process is being worked at a pilot plant scale before being

handed over to the industry. The number of research divisions and sections has remained the same and the research projects continue to be related to work on vegetable oils, surface coatings, fuels, drugs and pharmaceuticals, essential oils, entomology, biochemistry, inorganic chemistry, ceramics, physico-chemical, X-ray and instrumentation studies, chemical engineering, general engineering and operational research.

RESEARCH STAFF

Director

S. Husain Zaheer, M.A. (Oxon.), Dr.Phil.Nat. (Heidelberg) F.I.I.C., M.I.I.Chem.E.

Deputy Directors

S. A. Saletore, B.Sc. (Banaras), Ph.D. (Liverpool), A.I.I.Sc. (Bangalore), M.I.I.Chem.E., A.R.I.C.

Assistant Directors

D. S. Datar, B.A., M.Sc., Ph.D. (Bombay), A.I.I.Sc. (Bangalore M.I.I.Chem.E.

J. S. Aggarwal, M.Sc. (Hons.), Ph.D. (Punjab), Certificate in I and Drying Oils (U.K.)

G. S. Sidhu, B.Sc. (Hons.), M.Sc., Ph.D. (Lucknow)

M. G. Krishna, M.Sc. (Andhra), Ph.D. (Leeds), A.M.I.I.Chem.

G. S. Chowdhury, B.Sc.Eng. (Mech. & Elec.) (Banaras), A.M.I. (India), A.M.I.I.Chem.E.

K. S. Chari, B.Sc. (Madras), Ph.D. (Manchester), A.R.I.C., A.M.I.I.Chem.E.

Senior Scientific Officers, Gr. I

I. K. Kacker, B.Sc. (Hons.), M.Sc., Ph.D. (Lucknow)

K. T. Achaya, M Sc. (Madras), Ph.D. (Liverpool), A.I.I.Sc. (Bangalore)

C. Chenna Reddy, M.Sc. (Appl. Chem.) (Osmania), M.Ch.E. (I New York), Ph.D. (Ohio State), A.M.I.I.Chem.E.

Y. Venkatesham, B.Sc. (Madras), M.Sc. (Osmania), B.Ch.E. (I. Chicago), M.S., Ph.D. (Ohio State), A.M.I.I.Chem.E.

M. B. Naidu, M.Sc. (Aligarh), Ph.D. (London), F.R.E.S.

M. A. Sivasamban, M.Sc. (Appl. Chem.) (Osmania), A.M.I.I.Chem.E.

B. S. R. Sastry, B.Sc. (Madras), B.Sc. (Tech.) (Banaras), M.S. (Washington), Ph.D. (Penn. State)

K. G. Rangrez, M.Sc. (Appl. Chem.) (Osmania), M.Sc. (Tech.) (Manchester), A.R.I.C., A.M.C.T., A.M.I.I.Chem.E.

Senior Scientific Officers

L. M. Srivastava, M.Sc. (Osmania)

M. A. Hai, M.Sc. (Osmania)

G. S. Shenolikar, M.Sc. (Bombay)

S. S. Joshi, M.Sc. (Tech.) (Banaras), A.M.I.I.Chem.E.

K. N. Moorthy, M.Sc. (Banaras), Ph.D. (Osmania)

S. Raghavendar Rao, M.Sc. (Andhra), M.S., Ph.D. (Wisconsin)

Bharat Bhushan, B.Sc. (Hons.), M.Sc. (Punjab)

Baldev Singh, B.Sc. (Punjab), B.Pharm. (Banaras), A.I.Inf.Sc.

E. R Saxena, M.Sc. (Appl. Chem.), Ph.D. (Osmania), A.M.I.I.Chem.E.

B. S. Narayana Rao, M.Sc. (Tech.) (Banaras), A.M.I.I.Chem.E.

M. A. Wahab, M.Sc. (Appl. Chem.) (Osmania), M.S. (Ceram. Eng.) (Ohio State)

R. Vaidyeswaran, B.Sc., B.Sc. (Tech.) (Madras), A.M.I.I.Chem.E. (on leave in West Germany for higher studies)

R. J. Sujir, B.Sc. (Madras), M.Sc. (Bombay)

N. Sambasiva Rao, M.Sc. (Andhra), A.R.I.C.

Kumari Sardar Mahboob, M.Sc. (Osmania), Ph.D. (London)

P. M. Bhargava, M.Sc., Ph.D. (Lucknow)

D. P. Agrawal, B.Sc., B.Sc. (Tech.) (Nagpur)

B. S. Lulla, Ph.D. (Bombay), A.I.I.Sc. (Bangalore)

M. Ramacharyulu, B.Sc. (Hons.), M.Sc. (Andhra), Ph.D. (Bombay)

Junior Scientific Officers

V. S. Subrahmanyam, M.Sc. (Saugor), Ph.D. (Osmania)

N. Bhojraj Naidu, M.Sc. (Tech.) (Banaras), Ph.D. (Osmania)

Y. V. Subba Rao, M.Sc. (Appl. Chem.) (Osmania), Dip.German

Senior Scientific Assistants

G. Satyanarayana Rao, M.Sc. (Tech.) (Banaras), (on leave in U.S.A. for higher studies)

M. D. Narasimhan, B.Sc. (Madras), B.Sc. (Tech.) (Banaras), S.M., Sc.D. (M.I.T., U.S.A.) (resigned, 28–2–1959)

V. D. N. Sastri, M.Sc. (Hons.), D.Sc. (Andhra)

T. L. Narasimha Rao, B.Sc. (Madras), M.Sc., Ph.D. (Osmania)

Kumari Razia Osmani, M.Sc., Ph.D. (Osmania)

Shrimati Z. H. Osmani, M.Sc. (Osmania)

M. S. Kumaraswamy, M.Sc. (Appl. Chem.) (Osmania)

D. D. Jatkar, B.Sc. (Osmania), Cert. Paper Tech. (F.R.I., Dehra Dun)

V. P. Harigopal, M.Sc. (Tech.) (Osmania)

S. Zainul Abideen, M.Sc. (Appl. Chem.) (Osmania)

B. Rama Rao, M.Sc. (Banaras) (on leave in West Germany for higher studies)

P. E. Sattur, M.Sc., Ph.D. (Karnatak)

K. Seshagiri Rao, B.Sc., (Andhra), M.Sc. (Appl. Chem.) (Osmania)

G. Thyagarajan, M.Sc. (Osmania)

M. C. Menon, B.Sc. (Travancore), Dip. in Tech. (Oils, Soaps and Aromatics)

V. Krishnamoorthi, B.Sc., B.Sc. (Tech.) (Madras)

M. Noman Khan, M.Sc. (Tech.) (Osmania)

S. A. Qader, M.Sc. (Banaras)

Devendra Kishen Rao, M.Sc. (Appl. Chem.) (Osmania)

S. Neelakant Rao, M.Sc. (Appl. Chem.) (Osmania)

M. B. Narasimha, M.Sc. (Tech.) (Osmania)

S. Solayappan, B.Sc., B.Sc. (Tech.) (Madras), A.M.I.I.Chem.E.

Junior Scientific Assistants

S. Moinuddin Ahmed, M.Sc. (Osmania)

Mohd. Ehsan, M.Sc. (Osmania)

Mohan Lal, M.Sc., (Osmania)

H. Gopalakrishnan, M.A. (Madras)

S. Jaleel Hasan, M.Sc. (Osmania)

Kumari Safia Mehdi, M.Sc. (Osmania)

Kumari Salma Ansari, M.Sc. (Osmania)

K. Ranganathan, B.E. (Annamalai) (resigned, 21-6-1958)

K. M. Ghouse, M.Sc. (Osmania)

Mohd. Khursheed Hussain, M.Sc. (Osmania)

S. Anwarul Hasan, M.Sc. (Appl. Chem.) (Osmania)

G. Balamalliah, M.Sc. (Osmania)

K. M. Murad, B.Sc., B.Chem.Eng. (Osmania)

N. K. Sogani, M.Sc. (Rajasthan)

V. Krishna Rao, M.Sc. (Chem. Eng.) (Andhra)

M. K. Govind Rao, B.Sc., B.Chem.Tech. (Osmania)

K. G. Raghuveer, B.Chem.Tech. (Osmania)

S. Rajender Rao, M.Sc. (Tech.) (Osmania)

I. Burhanuddin, B.E. (Chem. Eng.) (Annamalai) (resigned, 28–2–1959)

Kedarnath Jape, M.Sc. (Osmania)

S. V. S. Kashmiri, M.Sc. (Lucknow)

S. N. Rajagopal, B.Sc., B.Chem.Tech. (Osmania)

T. Ranganathachar, B.Sc. (Madras), M.Sc. (Banaras)

C. George Kutty, M.Sc. (Appl. Chem.) (Kerala)

C. Narasimha Rao, B.Sc., B.Chem.Eng. (Osmania)

Senior Research Fellows

V. V. R. Subrahmanyam, M.Sc. (Tech.), Ph.D. (Osmania)

Shrimati Aziz Mirza, M.Sc., Ph.D. (Osmania)

Junior Research Fellows

T. P. Dharmarajan, M.Sc. (Appl. Chem.) (Kerala) (resigned, 30–4–1958)

G. M. Sastry, B.Sc. (Andhra), B.Sc. (Tech.) (Bombay)

Kumari A. Bhramaramba, M.Sc. (Banaras)

M. Janardana Rao, B.Sc. (Andhra), M.Sc. (Banaras)

K. A. Abraham, M.Sc. (Agra)

Mohd. Swaleh, M.Sc. (Aligarh)

B. Gopalakrishnamurthy, B.Sc. (Andhra), B.Chem.Tech. (Osmania)

G. Ramamoorthy, B.E. (Chem. Eng.) (Annamalai)

A. K. Jaiswal, M.Sc. (Osmania)

P. Radhakrishnamurthy, B.Sc. (Andhra), M.Sc. (Osmania)

Mohd. Abdul Jaleel, B.Sc., B.Chem.Tech. (Osmania)

Senior Laboratory Assistants

M. Chandra Sekhar

A. V. Venkat Rao, B.Sc. (Poona)

M. M. Hamza, B.Sc. (Osmania)

S. Akbar Azam, B.Sc.,LL.B.(Osmania)

S. Iqbal Ahmed, B.Sc. (Osmania)

Shrimati Shyamala Rao, B.Sc. (Osmania)

M. Waheeduddin, B.Sc., B.Chem.Tech. (Osmania)

S. M. Mohsin, B.Sc. (Osmania)

B. Gopinath, B.Sc. (Osmania)

M. Naseemul Huq, B.Sc. (Osmania)

S. Zainulabideen, M.Sc. (Osmania)

Junior Laboratory Assistants

M. Zulfiqar Ahmed, B.Sc. (Osmania)

Waheedududdin Ahmed, B.Sc. (Osmania)

M. Jafar Ali Khan, B.Sc. (Osmania)

Kumari Shanta Bai, B.Sc. (Mysore)

K. S. Moizuddin, B.Sc. (Osmania),Dip.Publ.Admn.

M. Waris Baig, B.Sc. (Osmania)

Ch. V. Suryanarayana, B.Sc. (Osmania)

V. Ramaswamy, B.Sc. (Osmania)

V. Raghunath Rao, B.Sc. (Andhra)

Jai Rao B. Kulkarni, B.Sc. (Osmania)

STAFF WORKING ON SPONSORED SCHEMES

New uses of Castor oil (Indian Central Oilseeds Committee, Hyderabad)

R. Subba Rao, M.Sc. (Osmania) Senior Scientific Assistant

Manufacture of 8-hydroxy-quinoline (Biological Products Private Limited, Bombay)

V. K. Venugopal, M.Sc. (Osmania) Junior Scientific Assistant

Bleaching Earths (Government of Mysore)

M. K. H. Siddiqui, B. Sc., B.Chem.Tech., B.Chem.Eng. (Osmania) Junior Scientific Assistant

Active Carbon Grade I (National Research Development Corporation of India, New Delhi)

K. Seshagiri Rao,B.Sc. (Andhra), M.Sc. (Appl. Chem.) (Osmania) Senior Scientific Assistant (27-9-1958 to 12-12-1958)

S. C. Venkata Subbiah, B.Sc., B.Sc. (Tech.) (Madras) Senior Scientific Assistant

S. Ali Abid Zaidi, B.Sc. (Osmania)	Senior Laboratory Assistant
M. M. Hasan, B. Sc. (Osmania)	Senior Laboratory Assistant
S. M. Mohsin, B.Sc. (Osmania)	Senior Laboratory Assistant (12-12-1958 to 19-1-1959)
M. Raza Ali, M.Sc. (Osmania)	Junior Laboratory Assistant

White Cement Project (National Research Development Corporation of India, New Delhi)

A. V. Rajeswara Rao, M.Sc. (Tech.) (Banaras), Ph.D. (Osmania)	Senior Scientific Assistant (till 4-12-1958)
K. Khaleeluddin, M.Sc. (Osmania)	Junior Scientific Assistant (resigned, 7-10-1958)

Separation of Silica from Green Liquor (Messrs. Orient Paper Mills, Brijrajnagar)

A. V. Rajeswara Rao, M.Sc. (Tech.) (Banaras), Ph.D. (Osmania)	Chemist

AUXILIARY TECHNICAL STAFF

Works and Maintenance

M. Guruswamy Naidu	Civil Engineer
S. A. Salam	Senior Technical Assistant
M. Murtuza Ali, B.Sc. (Osmania)	Head Laboratory Supervisor
Aijaz Ahmed	Chief Glass Blower
S. Hyder Ali Zaidi	Glass Blower
M. Namdar Ali Baig	Mechanic Grade I

Stores

A. Shanker, B.A. (Osmania)	Junior Scientific Officer
Asghar Husain, B.Sc. (Osmania)	Senior Store-keeper
Shaik Rasool	Senior Store-keeper

General Engineering

M. K. Chary, A.M.I.E. (India)	Senior Technical Assistant
G. C. Gopi Reddy, B.E. (Mech.) (Andhra), GRAD.I.E. (India)	Senior Scientific Assistant
S. H. Bilgrami, Dip. Faraday House (London), Certificate Marconi College, (Chelmsford, England)	Senior Scientific Assistant
M. L. Khosla, L.E. (Banaras)	Senior Draughtsman
A. Walter, L.M.E.E.	Senior Electrician
M. R. Muthyaloo	Fine Mechanic
Ahmed Ali Khan	Mechanic Grade I
Hifzur Rahman	Mechanic Grade I
B. V. Dasaratha Rao, L.M.E.E.	Mechanic Grade I
Sardar Khan	Mechanic Grade I
A. Ataullah	Mechanic Grade I
M. Shanker Rao	Foreman
A. M. Taj	Foreman
M. V. Raghavulu	Foreman

Library

Ahmed Sultan B.A. (Osmania), Dip.Lib.Sci., Dip. German, Dip.French	Senior Librarian
Anwer Hasan, B.A. (Osmania), Dip.Lib. (Bombay)	Junior Librarian
S. D. Bhogle, B.Sc. (Osmania), Cert.Lib.Sci.	Junior Librarian (resigned, 8—11—1958)
Kumari Shaher Bano, B.A. (Osmania), Cert. Lib. Sci.	Junior Librarian

ADMINISTRATIVE STAFF

M. C. Joshi, M.Sc.(Bombay), M.Sc (Tech.) (Manchester), A.M.C.T.	Administrative Officer
V. Sanker Narayanan, B.A , (Madras)	Accounts Officer
Rajendar Narain, M.Sc. (Agra)	Section Officer Grade II
C. S. S. Rao	Section Officer Grade III
M. A. H. Siddiqui, B.A. (Osmania)	Assistant
M. C. S Rao, B.A., LL.B. (Osmania)	Assistant
F. C. Chhabra, B.A. (Punjab), LL.B. (Agra)	Assistant

Sadananda Mehta, M.A., Dip. in Journalism (Punjab), LL.B., Dip. in Amer. Diplomacy and Int'l. Organisation, Proficiency in Russian language (Delhi),	Assistant
P. V. Rao	Senior Accountant
S. Mohiuddin	Junior Accountant
B. K. Dhanraj	Personal Assistant to the Director
Y. S. Rajlingam, B.Com. (Osmania)	Senior Stenographer
Shrimati Razia Sultana Osman	Receptionist

GENERAL ADMINISTRATION

Meetings and Committees

Executive Council : The 5th and 6th meetings of the Executive Council were held on September 13, 1958 and February 24, 1959, respectively. The Executive Council reviewed the progress of various research and pilot plant projects of the laboratory and the sponsored research schemes.

The Council accorded sanction for the purchase of equipment and apparatus at a total cost of Rs. 2,16,825 and the construction of the library building at an expenditure of Rs. 1.847 lakhs. The Council also accorded approval for the construction of Museum and Lecture Hall at an estimated cost of Rs. 2.71 lakhs, purchase of a new staff car, various items of services and furniture amounting to Rs. 3.06 lakhs, and the purchase of further items of equipment and apparatus during the year 1959–60 worth about Rs. 2.10 Lakhs.

The Council decided to provide working capital for running of the L.T.C. plant, and various minor projects for recovery of its by-products, as one integrated unit.

Scientific Sub-Committee : The Executive Council constituted a Scientific Sub-Committee to consider the programme of work, review the progress of research and make recommendations relevant to the implementation of research projects and pilot plant investigations, requirements of equipment, apparatus and buildings, scientific and technical personnel and such other matters referred to it by the Executive Council.

The sub-committee met twice during the year on September 12, 1958 and on February 10 and 11, 1959.

Experts Sub-Committee : An Experts Sub-Committee was constituted by the Executive Council with the Director as chairman, to consider departmental promotions and crossing of the efficiency bars. The committee met on February 9, 1959.

The personnel of the committees are given as Appendix.

Buildings

¶ The following works have been completed :

1. Three temporary sheds for storing bulk materials.
2. Muram roads in the laboratory premises.
3. Sanitary and drainage fittings to the Low-temperature carbonisation laboratory and the post office building.
4. O. C. B. Unit at the electric sub-station.
5. Laboratory and store room and platform for the fat splitting and fatty acid distillation pilot plant.
6. Compound wall.
7. False ceiling with plaster of Paris in the Director's office.
8. Septic tanks with soaking channels for the Low-temperature carbonisation laboratory and the post office building.
9. Ramp to the laboratory basement.
10. Cardboard ceiling for the Insect Room.
11. Approach road to the culvert near Low-temperature carbonisation pilot plant.

¶ The following items of works are in progress :

1. Construction of stores for inflammable materials.
2. Galleries to the pilot plant building.
3. Installation of low temperature room for the Biochemistry Division.
4. Construction of library building.
5. Boring of tube wells.
6. Laying of 2" thick cement concrete non-skid flooring on the platform of the fat splitting and fatty acid distillation pilot plant.
7. Construction of open masonry drains along the pilot plant buildings from the Administrative Block.

Conferences, Symposia, Exhibitions, etc.

¶ Members of the Laboratory staff participated in the following :

1. Defence Science Conference, New Delhi, April 1958.
2. Conference on "High - temperature kinetics," Massachusetts, June, 1958.

3. Fourth International Congress of Biochemistry, Vienna, September 1958.
4. Symposium on "Chemotherapy in bacterial and virus infections," Lucknow, October 1958.
5. Malaria and other arthropod-borne diseases sub-committee informal meeting, Delhi, November 1958.
6. Symposium on " The utilisation of vegetable oils, " New Delhi, November 1958.
7. General Conference of UNESCO, Paris, November-December 1958.
8. Symposium on " Cottonseed and its by-products ", Hyderabad, December 1958.
9. First Conference of Oilseed Research Workers, Chandigarh, December 1958.
10. 12th Annual Technical Meeting of the Indian Institute of Metals and the Conference of Metallurgists, New Delhi, December 1958.
11. Symposium on Electrolytic Cells, Karaikudi, December 1958.
12. Symposium on "Proteins ", New Delhi, January, 1959.
13. Indian Sceince Congress, 46th Session, New Delhi, January 1959, and the Symposia on " Clay minerals " and " Solid State Physics," Chemistry Section of the Indian Science Congress.
14. "India 1958" Exhibition, New Delhi, October 1958 to February 1959.
15. All India Industrial Exhibition, Hyderabad, January-February 1959.
16. 11th Annual General Meeting of the Indian Institute of Chemical Engineers, Delhi, January 1959.
17. Symposium on " Nature of Coal. ", Jealgora, February 1959.
18. Golden Jubilee Celebrations of the Indian Institute of Science, Bangalore, February 1959.
19. Conference of All India Paint Manufacturers Association, Kanpur, February, 1959.
20. Conference on Operational Research, New Delhi, March 1959.

Details of papers presented are shown separately under PUBLICATIONS AND PATENTS.

Awards and Distinctions

Deputation:

Dr. S. Husain Zaheer, Director, was nominated by the Government of India, as a member of the Indian delegation to the General Conference of UNESCO, held at Paris during November 4 to December 4, 1958.

Research Degrees:

1. Shri A. V. Rajeswara Rao, Senior Scientific Assistant, was awarded the Ph.D. degree of the Osmania University, Hyderabad, for his thesis entitled "Study of mineral constituents of Hyderabad clays by differential thermal analysis and other methods."

2. Shri M. D. Narasimhan, Senior Scientific Assistant, was awarded the Sc.D. degree of the Massachusetts Institute of Technology (U.S.A.) for his work on "Sintering in the presence of a liquid phase."

3. Shri B. S. R. Sastry, Senior Scientific Officer, was awarded the Ph.D. degree of the Pennsylvania State University (U.S.A.), for his thesis entitled "Phase equilibria and liquid immiscibility in the system Lithia–Boric–Oxide–Silica."

All India Industrial Exhibition, Hyderabad:

1. Shri Venkata Narasiah, Moulder, was awarded a prize and Merit Certificate by the All India Industrial Exhibition Committee, Hyderabad, for his invention of "Domestic Water Filter" for use in village homes.

2. The Laboratory participated in the "Best Inventions" contest and was awarded six prizes for the following inventions:

i. Rural vat for hand-made paper industry.
ii. Electrode holder for manual arc welding.
iii. Starter safety relay.
iv. Motor control relay.
v. Cyclo-Spiral filter.
vi. Auto speed control.

3. The stall of the Laboratory was awarded first prize for general appearance and maintenance, and second prize for demonstration.

"India 1958" Exhibition, New Delhi

The Laboratory participated in the "India 1958" Exhibition by setting up a stall in the Science Pavilion. Four models of pilot plants (including one working model) and twenty charts showing processes and projects of the Laboratory were displayed.

Recognition as centre for research leading to doctorate degree in science

The Laboratory has been recognised as a centre for research leading to doctorate (Ph.D. and D.Sc.) in science and technology of the following universities:

Osmania University, Hyderabad: Andhra University, Waltair; University of Poona, Poona; University of Kerala, Trivandrum; Muslim University, Aligarh: Banaras Hindu University, Varanasi; Calcutta University, Calcutta, and Nagpur University, Nagpur.

Membership of Committees

Dr. S. Husain Zaheer:

Member: Development Council for oil-based Industries (Soaps, Paints, and Plastics), Union Ministry of Commerce and Industry; Reviewing Committee of the Central Sericultural Research Station, Berhampur; Andhra Pradesh State Development Council and Standing Advisory Committee for large scale Industries for Andhra Pradesh; Executive Committee, World Federation of Scientific Workers; Council of the Indian Chemical Society; Council of the Indian Ceramic Society; Council of the Indian Institute of Chemists, India; Scientific Sub-Committee, Indian National Commission of the UNESCO; Chemicals Committee of the Indian Standards Institution; Experts Committee for Synthetic Oil Products, Government

of India; Committee to advise on matters relating to Oil Exploration, Production and Refining, Government of India; Development Council for Scheduled Industries engaged in the Manufacture and Production of Heavy Chemicals (Acids and Fertilizers); Joint Committee of the Indian Central Oilseeds Committee and the Council of Scientific and Industrial Research for research in vegetable oils; Joint Sub-Committee of the Development Wing, Ministry of Commerce and Industry, Government of India and the Council of Scientific and Industrial Research, and its Sulphur Sub-Committee; Sub-Committee on the Production of Terpene Chemicals from Turpentine, of the Council of Scientific and Industrial Research; Board of Directors, Messrs. Sindri, Fertilizers and Chemicals (Private) Ltd., Sindri; Selection Committees of the Muslim University, Aligarh and the Banaras Hindu University, Varanasi; Committee relating to All India Board of Studies in Chemical Engineering and Technology; Experts Committee on Biochemistry, U.G.C.;

Honorary Professor: Department of Chemistry, Muslim University, Aligarh.

Fellow: National Academy of Sciences.

President: Association of Scientific Workers of India.

Dr. S. A. Saletore

Member: Bleaching Earths Sub-Committee of the Indian Standards Institution; Technical Sub-Committee of the Indian Central Oilseeds Committee; Vegetable Oil Research Committee of the Council of Scientific and Industrial Research; Selection Committees of the Nagpur University, Laxminarayan Institute of Technology and Department of Technology,

Bombay University; Publication und Research Committee of the Oil Technologists Association of India, Kanpur.

Dr. D. S. Datar

Alternate Member: Bleaching Earths Sub-Committee of the Indian Standards Institution; New Delhi.

Dr. J. S. Aggarwal

Member: Lubricants Sectional Committee, Textiles Sizing and Finishing Materials Sectional Committee, and Petroleum Solvents Sub-Committee of Indian Standards Institution; Publication and Research Committee of the Oil Technologists Association of India, Kanpur.

Dr. B. S. R. Sastry

Member: Panel of Experts for Ceramics of the Chamber of Small Scale Industries, Hyderabad.

Dr. Y. Venkatesham

Member: Panel of Experts for Chemical Engineering of the Chamber of Small Scale Industries, Hyderabad.

Shri Baldev Singh

Member: Central Executive committee of the Association of Scientific Workers of India; Asian Regional Executive Committee of the World Federation of Scientific Workers; Editorial Board, Vijnan Karmee; Committee for Science and its Social Relations of the Indian Science Congress Association.

Dr. E. R. Saxena

Member: Editorial Board, Vijnan Karmee; Central Executive Committee of Association of Scientific workers of India.

Shri G. Thyagarajan

Member Asian Regional Executive Committee of the World Federation of Scientific Workers.

Appointments, Transfers, Resignations, etc.

Appointments : (a) The following appointments to the gazetted posts were made during the year.

Dr. J. S. Aggarwal	Assistant Director
Dr. C. Chenna Reddy	Sr. Scientific Officer, Gr. I.
Dr. Y. Venkatesham	do
Dr. M. B. Naidu	do
Dr. B. S. R. Sastry	do
Shri M. A. Sivasamban	do
Shri K. G. Rangrez	do
Dr. B. S. Lulla	Senior Scientific Officer
Shri D. P. Agrawal	do
Dr. M. Ramacharyulu	do
Dr. N. Bhojraj Naidu	Junior Scientific Officer
Shri Y. V. Subba Rao	do
Shri A. Shanker	do (Stores)

(b) The following Senior and Junior Research Fellowships were awarded.

Shrimati Dr. Aziz Mirza	Senior Research Fellow
Kumari A. Bhramaramba	Junior Research Fellow
Shri M. Janardana Rao	do
Shri K. A. Abraham	do
Shri Mohd. Swaleh	do
Shri B. Gopalakrishnamurthy	do
Shri G. Ramamoorthy	do
Shri A. K. Jaiswal	do
Shri P. Radhakrishnamurthy	do
Shri M. Abdul Jaleel	do

(c) Appointments to the following non-gazetted posts were made.

Senior Scientific Assistants 10; Junior Scientific Assistants 16; Senior Laboratory Assistants 6; Junior Laboratory Assistants 7;

Mechanics Grade I. 2; Mechanics Grade II. 6; Machine Operators 4; Laboratory Operator 1; Foreman 1: Mistries 4; Assistants 2; Upper Division Clerks 2; Lower Division Clerks 4; Senior Accountant 1; Senior Stenographer 1: Junior Librarian 1; Laboratory Attendants 4; Laboratory Bearers (skilled) 8; Laboratory Bearers (unskilled) 6; Library Bearer 1; Jamadar 1; Kamatee 1; Vatmen 4; Assistant Vatman 1; Peons 3; Farrash 1; Helper 1.

Retirement:

Shri M. Balayya	Clerk Grade I.

Transfer:

Shri V. Ramanathan, Senior Stenographer, transferred to Central Electrochemical Research Institute, Karaikudi.

Resignations:

Dr. M. D. Narasimhan, Senior Scientific Assistant; Shri I. Buruhanuddin, Junior Scientific Assistant; Shri K. Ranganathan, Junior Scientific Assistant: Shri S. D. Bhogle, Junior Librarian; Shri V. Subba Rao, Mechanic Grade II: Shri P. Janakiram Reddy, Lower Division Clerk: Shri Rafeeq Ahmed, Laboratory Bearer (skilled).

Obituaries:

Shri M. A. Aziz	Laboratory Bearer (skilled)
Shri Hasanuddin	Kamatee

Budget for 1958–59.

Regular budget	*Provision (In rupees)*	*Expenditure (In rupees)*
Capital grant (including provision for pilot plant projects)	8,53,600.00	9,13,615.19
Recurring grant	11,58,800.00	11,79,879.00
Fellowships		35,111.45

L. T. C. pilot plant project	*Receipts*	*Expenditure*
Sales proceeds of the L. T. C. products	4,01,566.73	3,92,520.40
Active Carbon Grade I (National Research Development Corporation of India, New Delhi)	31,750.00 (credited to the N.R.D.C.)	26,140.00
Income from securities	320.54	
Departmental Receipts		
Sales proceeds of Active Carbon Grade I (for the year 1957-58, realised in 1958-59).	7,331.00	
Sales proceeds of hand-made paper.	15,684.31	
Sales proceeds of chlorinated turpentine.	108.00	
Fee for analysis and testing.	3,425.00	
Miscellaneous income.	18,150.56	
Sponsored schemes		

The expenditure shown has been incurred on the following schemes.

White cement (National Research Development Corporation of India, New Delhi).	11,531.49

Fuller's earth (Government of Rajasthan).	16,391.31
Fuller's earth (Government of Mysore).	3,144.69
Storage of cottonseed (Indian Central Oilseeds Committee, Hyderabad).	1,386.52
New uses of castor oil (Indian Central Oilseeds Committee, Hyderabad).	5,567.31
Manufacture of 8-hydroxy - quinoline (Messrs. Biological Products Ltd., Bombay).	1,611.68
Separation of Silica from green liquor (Messrs. Orient Paper Mill Ltd., Brijrajnagar).	1,016.13

Tours and Visits

Dr. S. Husain Zaheer

April 1958, New Delhi: Defence Science Conference; 16th meeting of the Development Council for Scheduled Industries engaged in the manufacture and production of Heavy Chemicals (Acids and Fertilizers).

April-May 1958, New Delhi: Second meeting of the Committee to advise on matters relating to Oil Exploration, Production and Refining, Government of India.

May 1958, New Delhi: Inaugural meeting of the Development Council for Oil-based Industries (Soaps, Paints and Plastics).

May-June 1958, New Delhi: Selection Committee meeting (CSIR).

June 1958, Srinagar: Discussion with Kashmir Government regarding development of lignite deposits in the Kashmir State.

July 1958, New Delhi: Official work (CSIR); Calcutta: Paint panel meeting of the Development Council for Oil-based Industries; Sindri: Meeting of the Board of Directors, Messrs. Sindri Fertilizers and Chemicals (Private) Ltd.

July-August 1958, Bombay: Meeting of the Soap Panel of the Development Council for Oil-based Industries.

August 1958, Aligarh: University Selection Committee meeting.

September 1958, New Delhi: Selection Committee meeting, CSIR.

November-December 1958, Paris: General Conference of UNESCO, as a member of the Indian Delegation.

December 1958, New Delhi: Official work, CSIR; Banaras: Selection Committee meeting of Banaras Hindu University.

January 1959, Bombay: (i) Meeting of the Development Council for Oil-based Industries (Soaps, Paints and Plastics), (ii) Meeting of the National Research Development Corporation of India; Delhi: 46th Session of the Indian Science Congress; and meeting of the Experts Committee of U. G. C.

February 1959, Delhi: Meetings of the Board of Directors, Sindri Fertilizers and Chemicals (Private) Ltd.

March 1959, New Delhi: (i) Fifth meeting of the Committee on Utilisation of Food and Agricultural Wastes; and the meeting of Board of Directors, Messrs. Sindri Fertilizers and Chemicals (Private) Ltd.; (ii) Conference on Operational Research;

Calcutta : Meeting of the Committee relating to All-India Board of Studies in Chemical Engineering and Chemical Technology.

Dr. S. A. Saletore

November 1958, New Delhi: Meeting of the Joint Committee of the Indian Central Oil-seeds Committee and the Council of Scientific and Industrial Research.

Dr. D. S. Datar

January 1959, Delhi: 46th Session of the Indian Science Congress.

Dr. J. S. Aggarwal

November 1958, New Delhi: Conference of the Central Oilseeds Committee of the Council of Scientific and Industrial Research.

February 1959, Kanpur: Conference of All India Paint Manufacturers Association ;Calcutta: Paint Panel meeting of the Development Council for Oil-based Industries.

Dr. G. S. Sidhu

September 1958, Kaghaznagar: Study of the processes of manufacture at the Sirpur Paper Mills and M/s. Sirsilk Ltd., and to discuss problems for collaboration of work.

October 1958, Lucknow: Symposium on "Chemotherapy in bacterial and virus infections," at Central Drug Research Institute; Delhi: Visit to Shri Ram Institute for Industrial Research.

February-March 1959, Delhi: Visit to the D.D.T. Factory: Discussion at the Ministry of Defence, Govt. of India; and visit to Shri Ram Institute for Industrial Research.

Dr. M. G. Krishna

June 1958, Srinagar: Discussion with the Kashmir Government regarding development of lignite deposits in the Kashmir State.

February 1959, Jealgora: Symposium on "Nature of Coal" at Central Fuel Research Institute.

Dr. K. S. Chari

September 1958, Kaghaznagar: Study of processes of manufacture at the Sirpur Paper Mills and M/s. Sirsilk Ltd., and to discuss problems for collaboration of work.

Dr. K. T. Achaya

November 1958, New Delhi: Symposium on the "Utilisation of Vegetable Oils" sponsored by the National Institute of Sciences of India.

February 1959, Bangalore: Golden Jubilee Celebrations of the Indian Institute of Science.

March 1959, Delhi: Third meeting of the Soap Panel of the Development Council for Oil-based Industries.

Dr. M. B. Naidu

November 1958, New Delhi: Malaria and other arthropod-borne disease sub-committee informal meeting.

Shri M. A. Sivasamban

December 1958, Chandigarh: First Conference of Oilseed Research Workers.

Dr. Y. Venkatesham

January 1959, New Delhi: Eleventh Annual General Meeting of the Indian Institute of Chemical Engineers.

Shri Bharat Bhushan

November 1958, New Delhi: Malaria and other arthropod-borne disease sub-committee informal meeting.

Shri Baldev Singh

April 1958, New Delhi: Defence Science Conference.

July 1958, Lucknow: Visit to Central Drug Research Institute and Railway Testing & Research Centre; New Delhi: (i) Discussion with National Research Development Corporation of India; (ii) Development Wing, Ministry of Commerce and Industry; (iii) Visit to Shri Ram Institute for Industrial Research and the D.C.M. Chemical Work.

September 1958, Kaghaznagar: Study of the processes of manufacture at the Sirpur Paper Mills and M/s. Sirsilk Limited, and discussion on problems for collaboration of work.

January 1959, Delhi: 46th Session of the Indian Science Congress

Shri B. S. Narayan Rao

September 1958, New Delhi: Discussion with Central Road Research Institute regarding testing the road tar samples prepared at R.R.L.

January 1959, Delhi: 46th Session of the Indian Science Congress.

Kumari Dr. Sardar Mahboob

September 1958, Trivandrum & Ernakulam: Cinnamon leaf oil survey in various parts of the State (Tellicherry, Cochin, Anjerakandy, etc.).

Dr. P. M. Bhargava

August-September 1958, Vienna: Fourth International Congress of Biochemistry. Visited various research institutions on the Continent, doing work in biochemistry.

January 1959, New Delhi: Symposium on Proteins held under the auspices of Society of Biological Chemists, India.

Dr. V. V. R. Subrahmanyam

December 1958, Chandigarh: First Conference of Oilseed Research Workers.

Dr. M. D. Narasimhan

December 1958, New Delhi: Twelfth Annual Technical Meeting of the Indian Institute of Metals and the Conference of Metallurgists.

Fats and Oils

1. CASTOR

1. 1. Refining of castor oil

Object : Unlike other vegetable oils, the refining of castor oil generally involves no alkali treatment, but merely implies precipitation of mucilage by hydration and bleaching with earth. Moreover, expelled castor oil is generally of low free acidity and this is no drawback in a majority of its uses. Recently, however, a neutral oil has been demanded by the lubricants industry. For hydrogenation to a hard wax-like material also, a high degree of acidity would cause catalyst failure by metal soap formation. The growth of the solvent extraction industry will result in high-acid, coloured oils whose quality would always bear upgrading.

Previous work : Earlier literature references are scanty and usually to unavailable journals. Frequently the use of sodium carbonate and of refining in solvent is mentioned, but complete refining data such as losses, resultant oil colour and bleachability are rarely given. Since this work was substantially completed, Uruska, Mossakowska and Niewadomski (Oleagineux, 1958, *13*, 855) have published details of a method involving preliminary brine treatment of the oil, and its subsequent refining with potash on a couch of brine, which differs entirely from the procedure worked out at this laboratory.

Experimental work and conclusions : In one sample, reduction of free acidity from 3.76 to 0.3—0.8 per cent with corresponding refining losses varying from 4 — 1 per cent could be accomplished using sodium carbonate. No colour reduction accompanied refining, and the refined oil could not be bleached with active carbon and earth, probably because both crude and refined oils were already of a pale colour (Y+5R, Lovibond 1-inch cell, 13 units).

Applying the refining procedure to another darker oil of free acidity 6.4 per cent this could be brought down to 0.2—0.8 per cent with corresponding oil losses of 9.5—7.5 per cent. The refined oil

was easily bleached with earth and carbon mixture to a Lovibond colour of 20 units(Y+5R, 1-inch cell), which equals First Grade Indian Standards. Direct bleaching of the same castor oil without alkali-refining gave only a slight reduction, especially of the red component, showing that apart from reducing acidity, refining is also necessary to render the oil amenable to bleaching. Moisture content of the oil after refining was of the order of 1 per cent and soap content about 100 p. p. m.

Bench-scale trials were run twice on 5 lb. lots of the second castor oil of FFA 6.4 per cent. In each case the free acidity came down to 0.6 per cent with a refining loss of 13.5 per cent. This loss is some 4 per cent higher than that in the laboratory work, and is almost certainly due to the limitations of the apparatus used, which did not permit clear visual separation of the oil and aqueous phases. The refined oil colour (expressed as above) was 27, the bleach colour 20, the moisture content of the oil 0.7 per cent and the soap content from 40 — 100 p. p. m. The method is being patented. Refining in the presence of brine, as suggested in the recent method *(loc. cit.)*, gave high refining losses in our hands.

Future work: Experimental work is complete. The process is now to be utilised by industry.

1. 2. Hydroxylation studies on castor oil

Object : To produce from castor oil products with several hydroxyl groups, for further utilisation in surface coating formulations, for dehydration and as a chemical raw material.

Previous work : Scanlan and Swern established procedures for hydroxylation at a double bond using hydrogen peroxide - glacial acetic acid *in situ*, followed by strong alkali treatment (J. Amer. Chem. Soc., 1945, *67*, 1788), and for epoxidation using hydrogen peroxide-acetic anhydride (*ibid*, 1945, *67*, 412). Application of the hydroxylation procedure to castor oil, its mixed fatty acids and oleic acid gave the corresponding pure hydroxylated acidic products in 80-95 per cent yields (Annual Report 1957-58, p. 13).

Experimental work and conclusions : Attempts were made to prepare the hydroxy glycerides, which are potentially more valuable for utilisation than the acids. When peracetic acid prepared *in situ* by

way of acetic acid was used for the preliminary epoxidation, the intermediate compound obtained from castor oil was highly acidic and did not respond to ring-opening by various reagents; the intermediate compound using hydrogen peroxide-acetic anhydride reagent was however satisfactory. Ring-opening to the glycol was not successful with either aqueous or alcoholic sodium carbonate, but proceeded smoothly with 1 -3 per cent alcoholic sulphuric acid to yield 70 per cent of trihydroxy 'castor' glyceride.

Reduction of the ester of the above trihydroxy material to the corresponding alcohol did not occur by the Bouveault-Blanc procedure owing to the presence of several hydroxyl groups. However, when ricinoleyl alcohol (prepared from methyl ricinoleate by sodium reduction, vide Annual Report 1957-58, p.14) was used as starting material, the intermediate epoxy compound could be treated with strong alkali to give alcoholic tetrahydroxy compound in 81 per cent yield.

In utilisation studies, the coupling of this alcohol with linseed fatty acids was tried. The product obtained gave a film whose properties are being studied, while variations in the coupling procedure and materials are being tried.

Future work: To study the utilisation of the polyhydroxy compounds obtained in several ways.

Note : This work was conducted under a scheme of the Indian Central Oilseeds Committee, Hyderabad, sanctioned from January 1, 1958 to March 31, 1960.

1. 3. Surfactants from castor oil

Object : To study in general the possibilities of obtaining surfactants from castor oil. The immediate purpose is to study the sulphated salts of ricinoleyl alcohol for surfactant properties. The presence of a central hydroxyl group usually confers wetting properties and that of a terminal hydroxyl group, detergency. The existence of both in castor alcohols is likely to result in properties which may repay study.

Previous work : The method of Hansley (Industr. Engng. Chem., 1947, *39*, 55) was applied to castor oil and its esters (Annual

Report 1957-58, *P*-13). Reduction of methyl ricinoleate using an extra atom of sodium, *t*-butanol as reducing alcohol (in preference to *t*-amyl or *sec*.-butyl alcohols), xylene as solvent medium and rapid addition of ester in 4 minutes gave 86 per cent yields of the corresponding alcohol, which could be purified by distillation to give product analysing very close to theory.

Experimental work and conclusions : Various sulphating agents were studied for their action on model materials – castor oil, oleyl alcohol, ricinoleyl alcohol and lauryl alcohol. Use of oleum as a sulphating agent at room temperature (30° - 35°C.) led to greater double-bond attack than with conc. sulphuric acid. Chlorosulphonic acid gave high hydroxyl sulphations, with an optimum at 0. 25 molar excess of the reagent. Attack at the double bonds by the agents was more pronounced in sulphating the the fatty alcohols than castor oil.

The series of neutralised sulphated products from castor oil, and from oleyl, ricinoleyl and lauryl alcohols were comparatively evaluated for wetting and detergent properties with the following results, in which H means high, M medium and L low :–

	Turkey Red oil	Sodium oleyl sulphate	Sodium ricinoleyl sulphate	Sodium lauryl sulphate
Calcium tolerance	L	H	M	M
Wetting power (Draves - Clarkson)	M	M	H	M
Dispersing power (Fisher - Dott)	L	M	M	H
Foam test (Ross – Miles) very	L	M	L	H
Emulsification power at 32°C.	H	————Identical M————		
Surface tension of 0. 5% soln. at 32°C. in dynes/sq. cm.	51	40	41	36
Detersive power on soiled cotton at 0. 2% conc. (as % of standard alkyl aryl sulphonate, 2 successive washes)	5	99	84	88

It is concluded that ricinoleyl alcohol, except for low foaming power (which can be supplemented), is promising as a surfactant; its wetting power is particularly high.

Future work : (i) To study the surfactant properties of ricinoleyl alcohol sulphate with suitable builders, and (ii) to study on similar lines the preparation and properties of castor monoglycerides.

2. COTTONSEED

2. 1. Refining of cottonseed oil

Object : To suggest process condition for the refining of Indian cottonseed oils to light-coloured products, especially when these oils are old and colour-fixed by poor and long storage of seed (Subrahmanyam, Achaya and Saletore, Indian Oilseeds J., 1958, *2*, 23).

Previous work : An enormous literature, chiefly American, is available on the refining of cottonseed oil, but colour-fixed oils are not commonly encountered in America because of good seed-storage practice. A large amount of work over several years has been carried out at this Laboratory (see Annual Reports since 1953) covering various aspects of the problem. This mass of data has been evaluated during this year.

Summary of conclusions :

Several factors were shown to have no effect on the yield or colour of the refined oil produced. These may be summarised as follows :—

(a) No rapid increase in colour or refining loss occurred in the first few days after expelling.

(b) Cooling hot oil as it emerged from the exepeller did not effect its subsequent refining characteristics.

(c) Results of refining at various temperatures from 8° to 40°C. did not differ.

(d) Preliminary clarifications with aqueous sulphuric, citric and tartaric acids were without effect on subsequent refining loss or colour.

(e) Use of additives like common salt, alum, tartaric acid and lignin during refining were generally of little or no benefit by either the wet or the dry refining procedures.

(f) Refining losses by the wet refining method were perhaps slightly lower than by the dry method.

Certain positive conclusions were also drawn from the earlier refining data :—

(a) In refining old and colour-fixed oils, increasing the strength or excess of lye merely increased the refining loss without improving the colour.

(b) With fresh, non-colour-fixed, low-acid oils, the use of strong or excess lye caused marked colour improvement without much effect on refining loss.

(c) Addition of 2–10 per cent alcohol to the refining lye greatly reduced the colour of the resultant refined oil with an increase of some 1 per cent in refining loss.

(d) As the FFA of crude oil increased, the colours of refined and bleached oils began to converge, and at about 9–10 per cent crude oil FFA, the refined oil was nearly incapable of bleaching.

Future work : To study especially the refining of colour-fixed oils in various promising ways.

2. 2. Storage of cottonseed and cottonseed oil

Object : To disperse widely the results of work on this subject obtained at this Laboratory (see Annual Reports from 1954).

Previous work : The results have been published as a Final Report, available from the Secretary, Indian Central Oilseeds Committee, Gandhi Bhavan, Hyderabad, or from the Director of this Laboratory. Two research papers and one general paper have also been published. At the request of the Indian Central Oilseeds Committee, the work done under this head was also written up in popular style for translation into Indian languages and wide distribution by the Committee. The pamphlet suggests practices likely to lead to the production of oils of premier quality at all stages of

seed production e. g., in the *field*, protection of picked cotton from rain; in the *ginning factory*, avoidance of violent handling of seed bags and quick disposal to the oil mill. It was recommended that at the *oil mill*, seed should be tested for oil quality before purchase and so packed that poor-quality seed is crushed first; storage should be cool and dry, with at least a good roof and flooring. Storage of crude oil is not desirable, and plant refining should be preceded by laboratory tests to determine suitable conditions of refining. For fresh oils of good quality, an excess of strong alkali will yield excellent products, while for old and colour-fixed oils washing with weak lye to remove free acidity, followed by strong alkali refining at a high temperature, was recommended.

Future work : Since oil refining is now realised to be closely connected with storage practices, certain aspects of the latter are planned for study under the scheme on refining. Crude oil storage without colour-fixation, which is comparatively rapid, is also planned for study.

2. 3. Fatty acids from cottonseed foots by distillation

Object : To obtain light-coloured fatty acids from cottonseed oil-refining foots.

Previous work: Cottonseed foots constitute a major by-product of the oil refineries, and cannot be easily or directly utilised because of their dark colour. By distillation, however, light-coloured fatty acids can be obtained. Extensive data are lacking in conditions for processing and purifying commercial Indian soapstocks. Seventy one per cent yields of light-coloured acids were obtained by distillation with superheated steam (Bharat Bhushan, Phillips and Gulati, J. Sci. Industr. Res., India, 1953, *12 B*, 38). Alkali-graining and separation of the neat soap by centrifugation has been found to result in a marked reduction of the oxidised impurities of soapstock with a resultant increase in distillation yields (Keith, Blachly and Sadler, J. Amer. Oil. Chem. Soc., 1954, *31*, 298). In this Laboratory straight distillation of the crude acids obtained from a commercial soap-stock containing 40 per cent total fatty acids yielded 62 per cent (on the still charge of light-coloured acids at 230–80°C. and 2–3 mm. pressure (Annual Report 1957–58, p. 19).

Experimental work and conclusions : A stainless steel kettle of 2-litre capacity, with an open steam coil and a gate valve, was desig-

ned and fabricated in the laboratory to facilitate the acidulation and separation of crude fatty acids. The black crude acids containing about 70 per cent total fatty acids were distilled, both through a fractionating column and straight, to yield 60 — 65 per cent still charge of distilled acids (about 85—88 per cent on the total fatty acids present). The acids can be fractionally distilled as two cuts: about 15 per cent of the total crudes as 'solid' acids of I.V. 13 — 15 and about 60 per cent comprising the 'liquid' acids of I.V. 128 — 130. The two cuts can also be obtained by straight distillation, but the I.V. of the first cut is about 75. The distilled acids have a persistent burnt odour which, however, can be removed by deodorisation by conventional methods. At a pressure of 2 — 3 mm. the fatty acids distil over at vapourtemperatures of 170°–212°C.

Graining with salt does not appear to have any effect in reducing the oxidised impurities of soapstock.

Future work: To study (i) graining of soapstock with caustic alkali, (ii) treatment of soapstock with borax and alcohol, (iii) aeration of the crude black acids prior to distillation, and (iv) to establish correct conditions as a guide in pilot-plant distillation (see FATTY ACIDS PROJECT).

2. 4. Pilot-plant processing of cottonseed

Object: Study of the economics of cottonseed processing on a pilot plant scale, since few data regarding delinting, dehulling and expelling of Indian varieties of cottonseed and refining of the oil are available.

Previous work: From the analysis of data collected on 20-ton commercial lot of fuzzy variety of cottonseed processed in cooperation with a local firm it was found that the power requirement was disproportionately high and varied from 0.35 to 2.00 KHW per lb. when the amount of first cut lint was increased. With the increase in the quantity of linters cut per day the cost per pound decreased, but there was a deterioration in the quality. The cost of first cut and second cut linters and that of seed processing in the existing pilot plant for cleaning, delinting, dehulling and separation of meats and hulls, for 3.5 tons of seed continuously, was also estimated.

Experimental work and conclusions : In co-operation with a local firm 55 tons of commercial variety of cottonseed containing 80 per cent high lintered and 20 per cent low lintered seed were processed working continuously three shifts and the yields of various products were: meats 59.7 per cent, hulls 32.9 per cent, linters 3.6 per cent, oversize 0.3 per cent, cotton dust 1.1 per cent, extraneous dust (earth) 2.1 per cent and loss 0.3 per cent.

On the basis of data collected the cost for processing the cottonseed worked out to about Rs. 50/- per ton. The market study of the products is being done by the party.

Programme of work for processing cottonseed from Andhra Pradesh has been sponsored by the ICOC. Ten tons of American Parbahani (Adilabad) and 10 tons of Gaorani, 6 (Bhainsa) are being obtained for processing.

A disc mill, elevator, conveyor, oil sump, filter presses with pumps, etc., were installed and the necessary pipe connections were made. A trial run of the D. P. Expeller was done and found satisfactory. One lint cleaning machine and one lint baling machine are being procured. Continuous oil refining plant having a capacity of about 1,2 ton of oil per 8 hours is being procured.

Future work : Delinting of various types of seeds will be studied, and decorticated and hull and meat ratio will be found. Expression of oil by double and single screw expeller will be made to study the effect of different variables like cooking, drying, pH control, heating and cooling of shaft, etc.

Costing will be done at every stage.

2. 5. Refining of Indian cotton linters

Object : To refine indigenous raw cotton linters to cellulose of different specifications suitable for nitration, alkylation and acetylation.

Previous work : Various conditions viz. cleaning, scouring with alkali, souring with acid, bleaching and de-ironing, involved in refining of linters to nitrocellulose grade, were established. A typical progressive analysis of linters was also made.

Experimental work and conclusions: Conditions have been established for refining linters to different viscosity grade and to obtain high

viscosity and low viscosity bleached linters. Tentative conditions to prepare low viscosity CMC have been worked out. Linters have also been refined for the preparation of methyl cellulose.

Future work: The work has been completed and the process is ready for industrial exploitation.

3. FATTY ACIDS

3. 1. Crystallisation of tallow fatty acids

Object: To study on a pilot plant any technical difficulties in the working of the process finalised in the Laboratory for the crystallisation of the mixed fatty acids of Indian beef tallow into mainly saturated and mainly oleic acid (Subrahmanyam and Achaya, *J. Amer. Oil Chem. Soc.*, 1958, *35*, 467; C. S. I. R., *Indian Pat.* 62,263, 1957).

Previous work. Laboratory conditions for optimum crystallisation were: to use 80 - 85 per cent ethanol at—5° to 12.5°C. at a solvent acid ratio of 5 - 10 to yield 70 per cent saturated acids of I.V. 11 (containing 7.6 per cent unsaturates) and 30 per cent 'oleic' acid of I.V. 94 (containing 2 - 4 per cent of saturates) (Annual Report 1957 - 58, p. 26.)

Present work: Work on designing and fabricating a pilot plant of suitable capacity to operate the process worked out in the laboratory has been taken up.

Future work: As stated.

3. 2. Basic tallow studies

Object: Since an Indian tallow industry is likely to be based on goat and sheep tallows rather than on beef tallows because of Indian social conditions, fundamental studies on these products are desirable.

Previous work: One component fatty acid analysis only of each type of tallow is available, of which the older analysis does not list minor components (Dhingra and Sharma, *J. Soc. Chem. Ind.*, 1924, *43*, 216 T; Hilditch and Shrivastava, *J. Amer. Oil Chem. Soc.*, 1949, *26*, 1). In regard to another aspect of the present work, viz., the establishing of standard conditions of crystallisation of the mixed

fatty acids from polar solvents at available low temperatures, to replace the lead salt separation for routine laboratory component fatty acid analysis. Gunstone and Garton (*Biochem. J.*, 1953, *54*, 617) used 10 parts of methanol at 20°C., but did not try aqueous dilutions of other polar solvents like ethanol and acetone.

Experimental work and conclusions: One sample each of the fat-rich tissues ('charbi') from a goat and a sheep carcass was rendered in the laboratory with boiling water. The analytical characteristics of each fat were determined and the goat tallow (I. V. 35. 0, I. V. of m. f. a. 35. 2) was analysed by the standard lead salt–ester fractionation procedure to yield the following component fatty acid analysis:—

Myristic 5. 0, palmitic 36. 2, stearic 20. 4, tetradecenoic 0. 2, hexadecenoic 3. 8, oleic 31. 7, octadecadienoic 2. 3 and higher unsaturated acids 0. 4 (as per cent wt.).

Further work : The work reported has just commenced and will continue on the lines stated above.

3. 3. Fatty acid separation using urea adducts

Object : To improve the selectivity of urea adduct separations of fatty acid mixtures while maintaining simplicity of operation.

Previous work : Since 1952, urea adduct separations of different fatty acid mixtures have been studied in this Laboratory in various media – methanol, ethanol and as solid. Selectivity could be improved by a short–time adduction in the cold, and this technique was applied to safflower, linseed and cottonseed fatty acids (Subrahmanyam and Achaya. *J. Sci. Industr. Res., (India)*, 1957, *16 B*, 269; Annual Report 1957-58, pp. 26 - 27).

Experimental work and conclusions :

(a) *Recovery of urea and alcohol* : If the precipitated urea adduct, especially, and the solubles after alcohol removal, were decomposed with 10 parts of boiling distilled water, fatty acid recoveries were only about half, and persistent emulsions formed. A minimum of 1 per cent hydrochloric acidulation of the water was necessary for complete fatty acid liberation. The 80 per cent alcohol used was recovered in 90 per cent strength and in 85 per cent yield on an alcohol basis.

(b) *Resolution of safflower acids* : Addition of the acids to the urea slurry rather than *vice versa* gave a high recovery of linoleic acid of the usual 90 per cent purity (73 per cent, I. V. 169).

(c) *Resolution of cottonseed acids* : About equal weights of four fractions of I. V. 20, 90, 130 and 155 were obtained from cottonseed fatty acids using the following conditions :—

	Primary adduction	Precipitate re-adduction	Solubles re-adduction	
Parts of urea	3	3–2. 5	4	6
Parts of medium	8	15	8 or	8
Time of adduction, minutes.	15	15	15	5

(d) *Simplification of technique* : Attempts have commenced to readduct the primary fractions with the urea already present *in situ* without isolation of fatty acids, as this will greatly reduce time and costs in the pilot-plant work which is planned.

Future work : Laboratory work to improve selectivity and to simplify the process will continue; pilot-plant work on typical fatty acid mixtures is planned.

3. 4. Spectrophotometric standards

Object : To establish spectrophotometric standard values in the ultraviolet for ∝-elaeostearic acid.

Previous work : Extinction coefficients for linoleic and linolenic acids after alkali-isomerisation under standard conditions were earlier established. Preparation of pure ∝-elaeostearic acid from tung oil was also described (Annual Report 1957–58, pp. 27 — 28).

Experimental work and conclusions : Based on a large number of absorption determinations on several preparations of the pure acid, the extinction coefficients of pure ∝-elaeostearic acid in absolute ethanol at 2680 A and 2330 A, were fixed at 146.3 and 10.6 respectively.

For quick determination of the percentage of the acid in tung oil, an accurately weighed sample of 70 mg. of the oil is saponi-

fied by boiling for a few minutes with 5 drops (160 mg.) of standard 21 per cent potash — glycol reagent and 5 ml. of absolute ethanol, and the absorption determined after suitable dilution with absolute ethanol.

Future work : Work on the above, for determining spectrophotometrically, linoleic, linolenic, and α-elaeostearic acid.

3. 5. The fat tropicalisation phenomenon

Object : To examine with reference to sunflower seed oil the relative saturation observed in the fats of fish, animals and plants raised in the tropics over those of the same species from cooler climes, which, it is proposed to call the ' fat tropicalisation phenomenon '.

Previous work : Numerous instances to document the general observation have recently been reviewed (Zaheer and Achaya, *Die Nahrung*, 1958, *2*, 710). Oils from sunflower seeds grown in various parts of the world have been found to differ markedly in unsaturation.

Experimental work and conclusions: An examination of the oils from sunflower seeds grown in Hyderabad during the cool months of 1955, 1956, 1957 and 1958 from seed originally brought from California, U. S. A., indicated that variations in individual seed-heads were quite marked (I. V. 115 to 133) and that any gradual manifestation of the tropicalisation phenomenon could not be judged on mixed oil samples. The seeds appeared to store well, with no marked rise in oil acidity. The average analyses of thirteen seeds and seed oils and the mixed fatty acids of the latter (determined spectrophotometrically) were as follows :—

Seeds: Seed index 8.2, per cent moisture 7.0, kernel 53.0, oil in kernel 43.7 and oil in seed 21.9.

Seed oils: S. E. 297.1, I. V. 123.4, per cent FFA 5.2, peroxide value 9. 3.

Mixed fatty acids: I. V. 126.8, per cent saturated 15.4, oleic 29.3, linoleic 55.3.

The extraordinary variation in composition of the same species of oil is strikingly brought out by comparing the component fatty acids at three degrees of unsaturation:

I. V. of mixed fatty acids	95	119	148
Component fatty acids, per cent wt.			
Saturated	9	14	10
Oleic	72	41	14
Linoleic	19	45	76

The first oil is roughly comparable to a groundnut oil rather high in oleic acid, the next to cottonseed oil and the last to safflower oil. Marketing vexations consequent on this extreme variability can be minimised by indicating the I. V. on each lot of oil, from which the component acids may be fairly deduced, and the lots suitable for different end-uses chosen. The tropicalisation phenomenon is consistent with the production of a minimum of unsaturated acids for fluidising purposes only, or may simply indicate that the unsaturated acids are synthesised independently of the saturated ones which tend to remain constant.

Future work: Work on the above is complete. Other interesting or unusual fatty oils will be studied when occasion arises.

3. 6. Fat splitting and fatty acid distillation

Object: To split fats and obtain distilled fatty acids for various requirements of the Indian industry.

Previous work: A literature note and a plan of work on splitting of fats by high pressure steam without the use of catalyst have been prepared. Preliminary work on linseed oil was done to standardise the technique.

The necessary auxiliaries required for the 100 kg. per batch pilot plant for fat splitting and fatty acid distillation have been designed and are being procured.

Experimental work: Two stainless steel samplers of about 30 kg. per sq. cm. pressure were designed, fabricated in the Laboratory and

fitted to a two litre stainless steel autoclave. Linseed oil fatty acids are at present in good demand by the paint industry. Linseed oil was hence used for splitting purpose and pilot plant trials are planned as soon as it is installed.

For every run of the autoclave the required amounts of oil and water were added and heated at a constant pressure maintained by an automatic arrangement; the mixture was stirred at 43–44 strokes per minute by a magnetic stirrer. Experiments were conducted with oil to water ratio of 2:1 and 1:1 at 20, 25 and 30 kg. per sq. cm. pressure. Samples taken at regular intervals to study the progress of the splitting were analysed for their acid and saponification values, and the percentage of split was calculated for each sample using the relationship:

$$\text{split per cent} = \frac{\text{acid value}}{\text{saponification value}} \times 100.$$

The data for five runs are given below:

Run no.	oil : water ratio	pressure kg/cm²	temp. C.	split per cent	glycerine conc.
1	2 : 1	20	225	77. 32	17. 75
2	2 : 1	25	230	77. 10	15. 53
3	2 : 1	30	240	77. 73	15. 46
4	1 : 1	20	225	87. 94	10. 60
5	1 : 1	25	235	86. 90	9. 95

For a specified ratio of oil to water the rate of split appears to increase with pressure although the maximum equilibrium split in each case remains independent of pressure. This appears to be true in both the ratios of oil to water studied although the maximum equilibrium split with lower oil to water ratio is higher than the other.

Pilot plant equipment: A platform measuring 72' x 48' for the fat splitting and fatty acid distillation pilot plant which has been received, and the two store rooms for the accessories of the plant were constructed: the steel structure measuring 16' x 12' x 50' de-

signed and fabricated in the laboratory has been erected. The staging of the structure for the erection of the plant is in progress.

A water cooling tower having a capacity of about 5000 gallons per hour has been received and is erected over a sump having a capacity of about 1,000 gallons for storage of water. Raw oil tank, deodorised oil tank, sweet water tank, fuel oil tank and acid tank are being installed. Foundations for two boilers have been completed and the Wehrlwerke boiler is being installed. Attempts are being made for procuring stainless steel sheets to fabricate the soapstock processing plant.

1.5 tons linseed oil has been procured for the trial runs of the pilot plant.

Future work : Laboratory study of the effect (i) of pressure and the ratio of oil to water and (ii) of some additives such as zinc oxide, fatty acids, etc., to ascertain their efficacy on the rate of splitting and the maximum split. The work will then be extended to the cottonseed and other oils.

Trial runs of the pilot plant on its installation will be taken for splitting of linseed oil and distillation of linseed oil fatty acids. cottonseed soapstock will be processed later and light coloured mixed fatty acids will be obtained. Optimum conditions of working on a pilot plant will be established and the economic and market data will be collected.

3. 7. Survey and market data

Object : To obtain basic information on the production, and actual and potential needs of fatty acids in the country.

Previous work : Information in regard to oleic and stearic acids, and of the market for hard waxes in boot polishes, was reported last year (Annual Report 1957-58, p. 30 and p. 15 respectively).

Present data :

(a) *Tallow utilisation* : The price of Indian tallow is about 0.44 nP./lb. (Rs. 1000/ton), and that of imported Australian tallow only slightly higher. Since the poorest grades of fatty acids sell at about 0.57 nP. and the best at about Rs. 1.60 nP. per lb., and while the price of by-product glycerine is Rs. 2.08 nP. per lb., the split-

ting and distillation or crystallisation of tallow fatty acids appear to be economically feasible in the country.

(b) *Splitting and distillation data*: Of ten parties approached, three have so far replied. All of them have splitting capacities of 2-3 tons per charge per day. The splitting process used is either the Twitchell, pressure or enzyme, with the trend in modern plants to high-pressure uncatalysed methods. Of the firms which replied, one has a 0.5 ton/day batch distillation unit designed and fabricated by themselves, and another a modern 5 ton/day continuous plant. Since the figures so far obtained are only partial, actual production data cannot be estimated, though the production of split acids is naturally much higher than the distilled ones. It is also learned that three more modern splitting and distillation plants, each of capacity 10 tons/day, are in course of erection in the country. The industry in India therefore appears to be developing.

(c) *Linseed fatty acids*: The potential demand for this product in the country was sought to be estimated by questionnaires to individual paint firms. Of the eight units that have so far replied, only the larger ones require the material in quantities of 10-12 tons per year, of as light a colour as possible. Their requirements are all obtained from Indian suppliers, and at least two paint manufacturers produce their own. So far the split acids rather than the distilled product appear to be exclusively used, judged from specifications.

Future work: To complete the above phase of the survey, and to obtain similar relevant data on other types of fatty acids and mixtures.

Surface Coatings

4. DEHYDRATED CASTOR OIL (D. C. O.)

4. 1. Dehydration of castor oil

Object: The D. C. O. prepared by using a catalyst mixture comprising sodium bisulphate, sodium sulphite and sodium bisulphite, suffers from a defect that its films and those of its varnishes are not sufficientlv water resistant. Other catalysts for the dehydration of castor oil have, therefore, been tried to overcome this deficiency in the product.

Experimental work and conclusions : Laboratory experiments were carried out on the dehydration of 300 g. lots of low acid value pale coloured castor oil under reduced pressure at 240°–250°C. using catalysts (i) 1.5 per cent sodium bisulphate, (ii) 1.5 per cent sodium bisulphate + 0.4 per cent sodium bisulphite, (iii) 1.5 per cent sodium bisulphate + 0.5 per cent sodium sulphite, (iv) 1.5 per cent sodium bisulphate + 2.0 per cent calcium carbonate, (v) 1.5 per cent sodium bisulphate + 0.5 per cent zinc dust, (vi) 1.0 per cent phosphoric acid, (vii) 1.0 per cent boric acid, (viii) 0.5 per cent sodium bisulphate + 0.5 per cent boric acid, (ix) 1.0 per cent sodium bisulphate + 1.0 per cent naphthalene 2–sulphonic acid, (x) 0.3 per cent phosphoric acid, (xi) 1.0 per cent sodium bisulphate + 0.3 per cent phosphoric acid, (xii) 1.0 per cent sodium bisulphate + 1.0 per cent sodium biphosphate, (xiii) 2.0 per cent sodium bisulphate + 1.0 per cent sodium biphosphate. (xiv) 1.5 per cent sodium bisulphate + 0.4 per cent sodium biphosphate, (xv) 1.5 per cent sodium bisulphate + 1.0 per cent calcium sulphite, (xvi) 1.5 per cent sodium bisulphate + 0.5 per cent zinc dust (80 mesh), and (xvii) 1.5 per cent sodium bisulphate + 0.5 per cent vanadium pentoxide.

Films of the products prepared using phosphoric acid or its salts as catalysts generally dried after a long time or remained tacky; the materials were, however, pale in colour. Experiment (xvi) above gave D. C. O. with best water resistance.

Future work : (i) To try a mixture of 1.5 per cent sodium bisulphate + 0.5 per cent zinc dust + 0.1 or 0.2 per cent phosphoric acid for getting a pale coloured D. C. O. with good drying characteristics; (ii) To determine the conjugated diene acid in all the above specimens of D. C. O. by the U. V. spectroscopic method.

4. 2. Pilot plant production of D. C. O.

Object : To establish optimum conditions of processing monomeric and bodied D. C. O.; to try some catalysts found satisfactory in the laboratory studies on the dehydration of castor oil; and to supply samples to interested parties for their trials.

Previous work : Ten charges, 20 lb. each, of monomeric D. C. O. and a few charges of bodied D. C. O. were prepared during the previous year. The costing of the process was done and com-

municated to the National Research Development Corporation of India, New Delhi. (Annual Report 1957–58, p. 32).

Experimental work and conclusions : Thirteen charges, 20 lb. each, of monomeric D. C. O. prepared in the Kestner pilot plant using certain catalysts were found satisfactory in the laboratory studies. The viscosities of the product varied between 1.4–2.0 poises at 30°C. Use of 1.5 per cent sodium bisulphate and 0.5 per cent zinc dust (80 mesh) was found most effective for the preparation of monomeric D.C.O. One charge of the monomeric oil was further bodied to 22.4 poises. Its Lovibond colour was 5.0 Y + 1.1 R through 1 cm. cell.

The process has been leased out for commercial exploitation to M/s. Jaiyant Oil Mills, Bombay, by the National Research Development Corporation of India, New Delhi.

Future work : Quantities of D. C. O. required for the laboratory work will be prepared.

4. 3. Storage studies of D. C. O.

Object : D. C. O. when kept in tinned steel containers darkened on storage for a few months,even after calcium carbonate treatment. Studies were undertaken to find a suitable container to reduce darkening of D.C.O. on storage; alternatively, to find an additive for the monomeric D. C. O. so that it may not discolour on storage.

Experimental work and conclusions : Monomeric D. C. O was stored in ordinary tinned steel containers, tinned steel containers coated inside with polyvinyl butyral resin pretreatment primer, plain mild steel containers coated inside with the above pretreatment primer, and plain mild steel container treated with 4 molar solution of phosphoric acid. No significant improvement in darkening of colour was observed in the samples of D.C.O. stored in the above containers for six months. Only the D.C.O. stored in containers coated with pretreatment primer was less coloured than the others.

Future work : (i) The above studies will be continued: (ii) Addition of ethyl aniline phosphate to D. C. O. before storage will

be tried; and (iii) Studies on storage of D. C. O. will also be undertaken in containers of galvanised iron and those lined with epoxy resin and CNSL condensate compositions.

4. 4. Isomerisation of D. C. O.

Object: Isomerisation of various drying oils is known to increase their drying characteristics. Detailed studies on isomerisation of D. C. O. and to evaluate their properties as drying oils will be undertaken.

Present work: A preliminary experiment was carried out using 2 per cent anthraquinone at 270°–275° C. for $2\frac{1}{2}$ hours under reduced pressure. An increase in conjugation to 6 per cent in the treated oil was found to take place.

Future work: (i) Isomerisation studies of D. C. O. using various catalysts; (ii) Dehydration of D.C.O. *in situ* in the presence of various isomerisation catalysts.

4. 5. Modification of D. C. O. by styrention

Object: Fast drying, pale coloured products giving water resistant films are claimed to have been obtained from D. C. O. and other drying oils by treatment with styrene, a comparatively cheap material likely to be in commercial production in India in the near future. Work on modification of D. C. O. with styrene has been undertaken to study the various factors in the course of reactions.

Experimental work and conclusions: Monomeric D. C. O. was heated at 200 – 260° C. in the presence of varying quantities of styrene and an oxidation catalyst. Clear pale coloured products with fast drying characteristics were obtained using 35 – 45 per cent styrene in the D. C. O. The films were highly water resistant and almost colourless. The reaction in the presence of a turpentine fraction gave improved products and greater amount of styrene could be incorporated in the product without any loss in quality. The total time of reaction was 6 – 10 hours as against 20 hours reported by earlier workers. The yield of the product in various experiments varied between 93 – 100 per cent.

Future work: (i) Study on some other catalysts for styrenation reaction, and determination of various characterestics of these

products, (ii) preparation of bigger charges up to 1 kg. of the product in the laboratory for sending to some interested parties for trials.

4. 6. Modification of D. C. O. by treatment with hypochlorous acid and *tert*-butyl hypochlorite

Object: When D. C. O. is reacted with either hypochlorous acid or *tert*-butyl hypochlorite and later dehydrochlorinated, products resembling tung oil are reported to have been formed. Work has been undertaken to get quick drying and, if possible, tack free products from D. C. O.

Experimental work: Monomeric D. C. O. and castor oil were treated with sodium bicarbonate and chlorine which gave products having 12 — 22 per cent chlorine content. Estimation of chlorine in the products was carried out after hydrolysis with butanol in which required amount of sodium was dissolved. This reagent proved superior to alcoholic potash or sodium ethoxide for the estimation of chlorine in such compounds.

Future work: (i) Dehydrochlorination of products from D. C. O., and simultaneous dehydration and dehydrochlorination of products from castor oil will be tried by thermal and chemical means; (ii) chlorination of D. C. O. and castor oil will be carried out to different percentages, and the characteristics of these products after dehydration and dehydrochlorination, particularly for their efficiency in surface coatings, will be determined.

4. 7. Recin Varnishes

Object: Esterification of the hydroxyl group of ricinoleic acid in castor oil with various drying oil fatty acids has been reported to yield products of improved drying characteristics. Studies to obtain such products from castor oil by this reaction have been undertaken.

Experimental work: Castor oil treated with rosin was malenised, reacted with polyols and then dehydrated. Very pale coloured products with improved drying characteristics were obtained. The films of these materials were colourless, tack free and also very hard and flexible.

Future work: (i) Different quantities of the components will be used and the characteristics of the products obtained will be

determined. Attempts will be made to increase the water resistance of the films of these products. (ii) Larger amounts of some of the compositions will be prepared for sending to the interested parties for their trials.

5. VARNISHES

5. 1. Varnishes from D. C. O. and other drying oils using ester gum as resin

Object : To evaluate the use of monomeric and bodied D.C.O. in the preparation of ester gum varnishes and to compare their properties with those obtained from linseed and tung oils.

Previous work: Medium oil – length varnishes based on ester gum were prepared from various grades of D. C. O. and also from linseed oil. The properties of varnishes based on D. C. O. were superior to those based on linseed oil. Varnishes based on monomeric D. C. O. were in their turn superior to those based on ordinary and bodied D. C. O. (Annual Report 1957 — 58, p. 33).

Experimental work and conclusions: Short and long oil varnishes using ester gum have been prepared from monomeric D.C.O., linseed oil and tung oil. The observations on the characteristics of these varnishes are elaborated as follows: (i) The films of all the varnish samples dried to touch in 3 to 3½ hours and became tack free overnight. (ii) The short oil varnishes gave harder films (scratch test about 1000) than the long oil compositions (scratch test about 700). (iii) The films of long oil varnishes blushed less in water than those of short oil varnishes. (iv) Films of linseed and tung oil varnishes blushed comparatively less than those of D.C.O. varnishes. (v) Long oil varnish films were more susceptible to attack by 1 per cent caustic soda solution than short oil varnish films. (vi) D. C. O. varnish films were more resistant to alkali solution than those of linseed and tung oil varnishes. (vii) Films of the varnishes cooked to 10 — 12 inch string were harder and more resistant to water and alkali solutions than those of varnishes cooked to 2 — 3 inch string. (viii) All the varnish films were resistant to white spirit and lubricating oil, but slight softening took place on contact with petrol.

5. 2. Varnishes from D. C. O. and other drying oils using synthetic resins

Object: To study the comparative properties of short, medium and long oil varnishes prepared from monomeric D. C. O., linseed stand oil and tung oil using synthetic resins, *viz.*, modified phenolic resin, maleic resin and pure phenolic resin.

Experimental work and conclusions: The following quantities of resins and oils were used for different grades of varnishes:

	short oil	medium oil	long oil
Resin	100 g.	100 g.	100 g.
Oil	175 g.	250 g.	350 g.
Thinner	220 g.	280 g.	360 g.

Maleic resin M 4000 and modified phenolic resin R 710 (both of British Resin Products) and modified phenolic resin Biolac 1208 (Hardcastle Waud & Co.,) were used. Cobalt and lead naphthenates were used as driers to give 0.05 per cent cobalt and 0. 5 per cent lead as metals on the weight of oil in the varnish. The varnishes were cooked to 8 — 10 inch string.

In all nine maleic resin varnishes and twelve modified phenolic resin varnishes have been prepared. The maleic resin varnishes were found to be quite reactive and their medium and long oil compositions required a mixture of white spirit and turpentine (4 : 1) to keep in solution and avoid rapid skinning. Modified phenolic resin varnishes were easy to prepare and required white spirit as solvent.

Future work: (i) Tung oil varnishes from the above maleic and modified phenolic resins will also be prepared for comparison. (ii) Pure phenolic resin varnishes from monomeric D. C. O., linseed stand oil will be prepared. Similar type of work will also be carried out with epoxy resins. (iii) The physical properties of all the varnishes and their resistance to water, alkali solution, acid solution and various organic solvents will be determined.

5. 3. Outdoor exposure test on paints prepared from oleoresinous vehicles

Object: To find out comparative rate of disintegration, by exposure tests, of the films of paints prepared from ester gum

varnishes, and to evaluate the comparative suitability of various oils in preparing these resins.

Experimental work : Medium oil – length varnishes from monomeric and bodied D. C. O. and linseed stand oil using ester gum as resin, and white paints prepared using different pigments and talc as extenders, were applied on 12" × 12" mild steel clean and phosphated panels by two coat system. The panels were kept exposed vertically and at 45° angle facing south. The panels are being examined after definite intervals for gloss, colour change, checking and cracking, chalking, blistering, flaking, rusting and dirt collection.

Future work : Continuation of work on outdoor exposure tests on ester gum varnish paints and extension of these tests on paints prepared from (i) synthetic resins, D. C. O., linseed oil and tung oil varnishes, (ii) long oil alkyds obtained from safflower, linseed oils and D. C. O.

6. ALKYDS

6. 1. Alkyds from safflower oil

Object : To prepare medium and long oil alkyds from safflower oil; to study the compatibility of pthalic anhydride at various stages of monoglyceride formation and determine characteristics of the products obtained.

Previous work : A safflower oil alkyd of 50 per cent oil–length, using litharge as catalyst, was prepared by azeotropic distillation, followed by pilot plant trials. The characteristics of the products have been reported earlier.

A modified alkyd apparatus on the principle of Dean and Stark was set up (Annual Report 1957-58, p.35).

Experimental work and conclusions : Safflower oil alkyds, oil-length 50 per cent, viscosity nearly 22.5 poises and acid value 10.5, were prepared by monoglyceride method, using litharge as catalyst (0.15 per cent on the weight of glycerol). They were compatible with xylene, white spirit, cholorinated cashewnut shell liquid and chlorinated turpentine. Studies on the compatibility of phthalic anhydride

with monoglycerides indicated that the alkyds derived from low (20.3) and high (45.4) monoglyceride content gave, resins of low (8.85 - 10.75 poises) and high (22.5 poises) viscosity respectively.

A few 60 per cent oil-length alkyds were also prepared. Their films dried tack free in about five hours. The baked films gave scratch hardness 1400—1800 and exhibited good water resistance.

6. 2. Comparative evaluation of 66 per cent oil-length alkyds from safflower, linseed and monomeric dehydrated castor oils

Object: To compare the methods of preparation and characteristics of alkyds and paints prepared from long drying oils, particularly the ability to stand outdoor exposure.

Experimental work and conclusions: Conditions have been established to prepare low acid value resins of 66 per cent oil—length using different concentrations of litharge and cobalt naphthenate. Alcoholysis rates of safflower and linseed oils have been determined. In the alcoholysis of linseed oil both the catalysts required more time for the first stage of glycerolysis and esterification than that of safflower oil. In safflower oil runs 0.08 per cent cobalt naphthenate was found to be as good as 0.15 per cent litharge. In the glycerolysis of monomeric D. C. O. large volume of water vapour contaminated with acrid vapours was evolved. Slight haziness in the case of some alkyds was removed by adding triphenyl phosphite. The films of these alkyds dried to touch in 2 1/2 to 3 hours. The baked films gave scratch hardness of about 1,000 g.

Two pilot plant trials at different bodyings of the glycerides of safflower oil were also prepared.

Future work: Preparation of alkyds of 66 per cent oil – length from the drying oils, *viz.*, safflower, tobacco seed, linseed and monomeric D. C. O., and of white paints from these alkyds; determination of their characteristics and their outdoor exposure tests.

7. EMULSION PAINTS

7. 1. Preparation of oleoresinous emulsion vehicles

Object: The emulsion paints are mostly derived from polyvinyl chloride copolymers, other synthetic resins and rubber latex

which are not produced at present in the country. Work has therefore been undertaken on the preparation of emulsion paints from oleoresinous compositions. This requires stable oleoresinous emulsion vehicles.

Previous work: Preliminary work has been done on the preparation of oleoresinous emulsion vehicle by adding ammonia and water to oleoresinous compositions. The emulsions were stable in the beginning but, ultimately, they broke up by losing ammonia gradually (Annual Report 1957 - 58, p. 34).

Experimental work and conclusions: The water phase consisted of many additives, mostly from the class emulsifying agents, bodying agents, preservatives, antifoaming agents and some alkali salts. The emulsifying agents consisted of inorganic and organic bases, proteins non - ionic ester type surfactants and sulphonated and sulphated surface actants. Aqueous phases consisting of casein or groundnut protein, sorbitol mono - oleate and alkali salt of alkyl sulphonate, gum arabic, phenol, pine oil and ammonia, to keep pH 8.8 — 9.2, behaved most satisfactorily. This solution was stirred and an equivalent quantity of medium oil - length D. C. O. ester gum varnish (95 per cent non - volatile) was mixed and homogenised at a fixed speed and for a definite time. The emulsions prepared either from casein or groundnut protein were quite stable when kept for two months at 50°—52° C. This accelerated storage period would be equivalent to storage for one year at room temperature.

7. 2. Preparation of alkyd emulsion vehicles.

Object: Resins of high acidity will lead to increased formation of ammonium or alkali salts of acids resulting in decreased water resistance property of the emulsion paint film. Attention is therefore directed to low acid value alkyd resins of long oil - length for the preparation of emulsion vehicles.

Experimental work and conclusions: A conventional 66 per cent long oil - length alkyd of acid value 6—7, prepared from safflower oil, phthalic anhydride and glycerine was used in the preparation of emulsion vehicle, by a method similar to that employed in preparing oleoresinous emulsion vehicle. With this alkyd, other additives in aqueous phase being the same as in oleoresinous emulsion vehicles, much larger amount of casein (minimum 10 per cent) was required for producing a stable emulsion. Only 5 per cent groundnut protein was

however necessary. The casein emulsion was almost milk white whereas the one from groundnut protein was yellowish in appearance. A white emulsion similar to that from casein could be produced using 5 per cent gelatine and the product remained stable in the accelerated storage test for more than two months. The results of photomicrographic studies of these three emulsion vehicles are shown below :

Emulsion vehicle	Size of the particles	Distribution of the particles in aqueous phases
10 per cent casein	60 per cent 1- 2 μ	uniformly distributed
5 per cent groundnut protein	30 per cent 5–10 μ 30 per cent 10–15 μ	a tendency to cluster together.
5 per cent gelatine	60 per cent 2- 3 μ	uniformly distributed

A double coating of each of the three emulsions was given to three asbestos cement panels at a 4 - hour interval and left for drying. All the films dried hard overnight. The brushing of casein and gelatine emulsion vehicles was smoother than that of groundnut protein.

A preliminary attempt was made to prepare in a pebble mill an emulsion paint from gelatine vehicle using titanium dioxide and talc as pigments. The paint was quite stable in an accelerated storage test and gave a uniform film when applied on asbestos cement panel.

Future work : Determination of (i) various characteristics of the above emulsion vehicles, (ii) stability and other characteristics of other emulsion vehicles prepared by substituting different additives than those employed earlier.

Preparation of (i) paints from the emulsion vehicles proved satisfactory, (ii) special type of alkyds in which the emulsifiers are built *in situ* and using them to prepare emulsion vehicles and emulsion paints.

It is intended to avoid proteins in the above formulations.

8. CASHEWNUT SHELL LIQUID (CNSL)

8. 1. Bobbin enamels from CNSL

Object: To prepare from indigenous cashewnut shell liquid an enamel composition for textile bobbins suitable to withstand hard wear, wetting, steaming, etc., without undergoing any deterioration, and comparable to the imported material.

Previous work: Cashewnut shell liquid, a raw material available in large quantities in India, was polymerised to various consistencies. After thinning with white spirit it was coated by dip method on bobbins and baked at 60° — 70° C. Even after 6 — 8 hr. heating under these conditions the films remained tacky (Annual Report 1957 — 58, p. 37).

Experimental work and conclusions: A resinous product was obtained from treated cashewnut shell liquid. After removing the water of reaction a special type of oleoresinous varnish was added followed by suitable driers and lamp black pigment. The composition was applied on the bobbins by three coat system, each coating being baked at 60° — 70° C. for six hours. When the treated bobbins were kept in boiling water for two hours, the increase in their weight was not more than 2 per cent. The films of the enamel composition remained glossy and did not develop cracks or any other deformation. A five gallon sample of this enamel compositoin supplied to a local bobbin manufaturer is on large scale tests.

Future work: (i) Studies on preparation of improved compositions at reduced cost, and determination of the characteristics of their varnishes, (ii) to find different uses of the varnishes or enamels and paints prepared from these compositions.

8. 2. Analysis and refining of CNSL

Object: To analyse the cashewnut shell liquid extracted indigenously by different methods and to upgrade it to the Indian Standards Specifications or the purchasing specifications of British Resin Products, U. K., and Irvington & Insulating Company, U.S.A.; and to eliminate the toxic nature of the material, which hinders its export to other countries.

Experimental work and conclusions : Eleven commercial samples of CNSL have been analysed. Eight different methods of upgrading of those which did not come up to the standard, especially the oven test, have been tried. A simple method of improving low grade CNSL by treating with a mixture of sulphuric and hydrochloric acids has been developed.

Future work : (i) Analysis and upgrading of some more samples of CNSL; (ii) preparation of cardanol from CNSL and utilisation of CNSL and cardanol in the preparation of phenolic resins and other surface coating materials; (iii) some fundamental work on CNSL and cardanol will also be planned.

8. 3. Market data on cashewnut shell liquid

Object : To study the position of the CNSL extraction industry in India and the utilisation of CNSL.

Present work : The following data were collected :

(i) *Production* : The average annual production in the country is estimated between 7,000 and 9,003 tons (Cashew and Pepper Export Promotion Council, Cochin).

(ii) Exports are given below :

Year	Quantity exported (tons)
1952	3,654
1953	5,033.50
1954	4,525
1955	5,078.25
1956	4,423
1957	4,951

(iii) *Prices* : Crude or untreated oil Rs. 800/ton.

Treated oil Rs. 1200/ton.

(iv) Data regarding consumption or utilisation of CNSL by various industries in the country, particularly by the Paints industry, is not available.

Future work: Assessment of the indigenous utilisation aspects of CNSL.

Organic Chemistry, Drugs& Pharmaceuticals

9. POTENTIAL PHARMACOLOGICAL COMPOUNDS

9. 1. Synthesis in the quinoline series

Object: To synthesise a series of heterocyclic compounds of the quinoline series *viz.*, substituted hydrocarbostyrils, carbostryrils and 1, 2, 3, 4—tetrahydroquinolines, and to evaluate them for pharmacological activity.

Introduction and past work: Many compounds in the quinoline series are widely used in medicine, particularly as antimalarials and antibacterials.

Synthesis of the following hydrocarbostyrils was completed starting from appropriately substituted nitro methoxy benzaldehydes:

8—methoxy 3, 4—dihydrocarbostyril,

8—hydroxy 3, 4—dihydrocarbostyril,

7—methoxy 3, 4—dihydrocarbostyril,

7—hydroxy 3, 4—dihydrocarbostyril, and

7—methyl 3, 4—dihydrocarbostyril.

The hydroxy compounds were obtained by the demethylation of the corresponding methoxy compounds.

7—Hydroxy 3, 4 — dihydrocarbostyril and 7 — methyl 3, 4—dihydrocarbostyril which synthesised unequivocally in the present work, helped to establish the identity of some of the compounds reported but not described by Meyer (*Ber.* 1927, *60*, 861). Several new intermediates, not known in literature, were also prepared (Annual Report 1957-58, p. 37).

Experimental work: (1) (a) 6—Methoxy 3, 4—dihydrocarbostyril was synthesised starting from 2-nitro 5-methoxy benzaldehyde as described in literature.

2—Nitro 5—methoxy benzaldehyde — ▷

nitro cinnamic acid — ▷ amino cinnamic acid

| | |
v v

nitro cinnamic ester — ▷ amino cinnamic ester

— ▷6 —methoxy dihydrocarbostyril.

In the same way starting from 2—nitro 5—hydroxy benzaldehyde, 6—hydroxy dihydrocarobstyril was prepared. The latter was also obtained by demethylation of the 6—methoxy compound. The mixed melting point was undepressed.

Three new carbostyrils namely 7—methoxy carbostyril, 7—methyl carbostyril, and 8—methoxy carbostyril, were also prepared by cyclisation of the respective *o*—amino cinnamic acid.

(b) Introduction of the basic side chain — $CH_2 — CH_2 — N(C_2H_5)_2$ *in hydrocarbostyril at position 1*: Hydrocarbostyrils behave like amides and do not show basic properties. Hence N—alkylation proceeds, if at all, only in poor yields. Direct condensation of hydrocarbostyril with 2—diethylaminoethylchloride did not proceed. Condensation by way of the sodium salt gave a product which is being characterised.

(c) 1, 2, 3, 4 — Tetrahydroquinolines : 3, 4 — dihydrocarbostyril on reduction with lithium aluminium hydride in tetrahydrofuran gave 1, 2, 3, 4—tetrahydraquinoline in 70 per cent yield. Preliminary studies on the reduction of the other substituted hydrocarbostyrils were also made.

(d) N—Substituted tetrahydroquinolines: Condensation of 1, 2, 3, 4—tetrahydroquinoline with 2—diethylaminoethyl chloride proceeded smoothly to give N—(2—diethylaminoethyl) 1, 2, 3, 4—tetrahydroquinoline, p.b. 155°/3 mm., hydrochloride m.p. 170° C.

(2) 2—Bromo 3—methoxy cinnamic acid : As reported previously the bromo acid obtained from the corresponding amino acid by Sandmeyer reaction did not analyse correctly. It was now prepared from 2—bromo 3—methoxy benzaldehyde obtained by a new route.

(3) U.V. and I.R. absorption spectra of carbostyrils and hydrocarbostyrils : The ultraviolet absorption spectra of carbostyrils and 3 : 4—dihydrocarbostyrils were determined and compared in collaboration with the Physics Department, Osmania University, Hyderabad. The spectra of eleven substituted carbostyrils and 3 : 4 dihydrocarbostyrils were recorded. The results support the view that carbostyrils exist preponderantly in the lactam form like the hydrocarbostyrils. Some other correlations were also attempted.

The ultra-violet absorption spectra readings of carbostyril, hydrocarbostyrils and some derivatives in absolute ethanol are tabulated below. The compounds marked with an asterisk were synthesised in the Laboratory.

Compound	λ max (m μ)					log Em				
carbostyril	215			270	325	4.60				
carbostyril				270	325					
* 7-methoxy carbostyril	222	223		280	325 340	4.37	4.41		3.85	4.17 4.06
8-methoxy carbostyril	218	233	256	282	336	4.29	4.45	4.47	4.04	3.68
3 : 4-dihydrocarbostyril			252					4.09		
3 : 4-dihydrocarbostyril	221		254		320 330	3.96		4.23		2.67 2.65

Compound	λ max (mμ)			log Em		
* 7–methyl–3 : 4–dihydrocarbostyril	221	254	336	3.87	3.80	2.48
* 7-hydroxy-3 : 4–dihydrocarbostyril		254 286 294			3.96 3.63 3.59	
* 7-methoxy-3 : 4–dihydrocarbostyril		252 285 292 325			3.61 3.30 2.96	
* 8-hydroxy-3 : 4–dihydrocarbostyril		250 291			3.99 3.76	
* 8-methoxy-3 : 4–dihydrocarbostyril		252 290	340		3.95 3.64	2.55

Future work : (i) Synthesis of 5-methoxy and 5-hydroxy 3, 4-dihydrocarbostyrils, (ii) reduction of the various substituted hydrocarbostyrils synthesised, to their corresponding tetrahydroquinolines by lithium aluminium hydride, (iii) introduction of basic side chains into the tetrahydroquinolines thus obtained, at position 1, and (iv) to continue efforts to introduce side chains directly into hydrocarbostyrils at the 1-position.

The compounds thus obtained will be evaluated for their pharmacological activity.

9. 2. Synthesis of isoquinolines

Object : To synthesise a series of 6 alkoxy, 5 : 6-dialkoxy, 5 - Cl - 6 - alkoxy and 6 : 7 - dialkoxy isoquinoline derivatives and evaluate them for analgesic, antispasmodic properties.

Experimental work and conclusions :

(1) Chlorination of m-hydroxy benzaldehyde :

Direct chlorination of *m*-hydroxy benzaldehyde (Hodgson and Beard, *J. Chem. Soc.*, 1926, 149) did not always give reproducible results. 2-Chloro 3-hydroxy benzaldehyde was therefore obtained by chlorinating *m*-hydroxy benzaldehyde with *t*-butyl hypochlorite. 2-Chloro 3-methoxy-*B* - phenylethylamine required as an intermediate was obtained by the following route :

3–hydroxy benzaldehyde —▷ 2 - chloro 3 - hydroxy benzaldehyde —▷ 2–chloro 3 – methoxy benzaldehyde —▷ 2 – chloro – 3 – methoxy cinnamic acid —▷ 2–chloro 3 – methoxy – *B* –phenyl propionic acid —▷ the amide —▷ 3 - methoxy 2–chloro - *B* - phenylethyl amine.

The synthesis proceeded smoothly up to the penultimate stage but the Hoffmann's degradation gave poor yield. The route through the reduction of the suitably substituted nitrostyrenes was chosen. The required 2 - chloro 3 - methoxy - ω - nitrostyrene was obtained by condensing 2 - chloro - 3 - methoxy benzaldehyde with nitromethane in the presence of acetic acid and ammonium acetate. It was converted to the corresponding *B* - 2 - chloro 3 - methoxy phenylethylamine by catalytic hydrogenation using different catalysts. Reduction with lithium aluminium hydride is also being studied. The hydrochloride salt of the amine thus obtained is being studied for its pharmacological properties at the Osmania Medical College, Hyderabad.

(2) Isoquinolines : It is intended to prepare (i) 5 - chloro 6 - methoxy 1, 2, 3, 4 - tetrahydro isoquinoline, (ii) 5 - chloro 6 - methoxy - 1 (substituted phenyl) 3, 4 - dihydro isoquinolines, (iii) 5 - chloro 6 - methoxy - 1 - (substituted benzyl) - 3 : 4 - dihydro isoquinolines.

(3) Tetrahydroisoquinolines : 2 - chloro 3 - methoxy - *B* - phenylethylamine has been subjected to Pictet - Spengler reaction to give 5 - chloro 6 - methoxy 1, 2, 3, 4 - tetrahydroisoquinoline.

Future work; To prepare 2 - chloro 3 - ethoxy and 2 - chloro 3 -methoxy - *B* - phenylethylamines and to convert them to the corresponding isoquinolines.

9. 3. Nitrostyrenes

Object : To study the fungicidal activity of ω - nitrostyrenes which are obtained as intermediates in the synthesis of *B* - phenylethylamines.

Experimental work : The following new nitrostyrenes were prepared :

2 - chloro 3 - hydroxy - ω - nitrostyrene,

2 - chloro 3 - ethoxy - ω - nitrostyrene,

2 : 6 dichloro 3 - methoxy - ω - nitrostyrene,

2 - nitro 3 - methoxy - ω - nitrostyrene,

2 - nitro 4 - methoxy - ω - nitrostyrene, and

6 - nitro 3 - methoxy - ω - nitrostyrene.

The following known nitrostyrenes were also obtained : 2-chloro, 3-methoxy and 2-hydroxy 3-methoxy - ω - nitrostyrenes. 2-Iodo 3-methoxy benzaldehyde was prepared by the mercuration and subsequent iodination of *m*-hydroxy benzaldehyde by the method of Lock & Notts, followed by methylation. This was converted to its ω - nitrostyrene. Similarly 2 - bromo 3 - hydroxy benzaldehyde was obtained by the reaction of bromine in acetic acid on 2-mercuroacetoxy 3-hydroxy benzaldehyde. The bromo benzaldehyde can otherwise be prepared only through the corresponding amino aldehydes which are themselves difficult to prepare. Methylation of 2-bromo 3-hydroxy benzaldehyde gave 2-bromo 3-methoxy benzaldehyde.

Nitrostyrenes from 2 - iodo 3-hydroxy and 2-bromo 3-methoxy benzaldehydes did not give correct analytical results.

Evaluation of these nitrostyrenes for fungicidal activity against *Aspergillus niger* showed that 2-iodo 3-methoxy nitrostyrene was active in dilutions of one in 100,000. All the nitrostyrenes were active in dilutions of one in 10,000 except, the dichloro compound.

Future work: To study the effect of - *B* - methyl group on the fungicidal activity, i. e., to synthesise a variety of substituted *B* - methyl—ω—nitrostyrenes of the formula $R - CH = \underset{\displaystyle CH_3}{\overset{\displaystyle |}{C}} - NO_2$

10. PHENYL ACETIC ACID

Pilot plant production

Object and introduction: Phenyl acetic acid and phenyl acetamide are used as promoters in the fermentative production of penicillin. The main consumers in India are Hindustan Antibiotics (Private) Ltd., Pimpri, whose annual requirements of the acid and amide are fifty two and thirty two tons respectively, valued together at approximately rupees one million. The entire quantity is at present imported. Phenyl acetic acid and allied chemicals are also extensively used as perfumery materials.

Previous work: Bench scale preparation of these chemicals was completed (Annual Report 1956 - 57, pp. 31 - 32). Based on these yields obtained, projects for the production of these chemicals on a commercial scale were compiled. Economic data and costing were worked out for pilot plant and commercial units each with a capacity of 3 cwt., 0. 5 ton and 2 tons per day. Contact was established with industrial parties likely to be interested and technical information supplied to them.

Experimental work: (i) Conditions were established for the chlorination of toluene to benzyl chloride. Four batches of toluene (20 - 25 kg. each) were chlorinated and fractionally distilled. The first batch was spoiled and resulted mainly in nuclear chlorination. The second batch gave only 70 per cent yield of benzyl chloride while the third and fourth batches gave an yield of 86 per cent each.

(ii) Conversion of benzyl chloride to benzyl cyanide was carried out in 5 - 6 kg. batches. Conditions have been established to get yields of about 85 per cent.

Future work: To work out optimum conditions for the hydrolysis of benzyl cyanide to phenyl acetic acid and phenyl acetamide on pilot plant scale.

11. LEVULINIC ACID

Pilot plant production

Object : Calcium levulinate is a valuable pharmaceutical used in calcium therapy. Utilisation of molasses to produce levulinic acid would lead to more economic method for the production of this pharmaceutical.

Previous work : Shanmukha Rao, Ramachandran and Zaheer *(J. Sci. Industr. Res., (India)*, 1953, *12 B*, 188) worked out a process for the preparation of levulinic acid from sugar-cane molasses, giving yields up to 45 per cent of the acid based on sugar present in molasses. A simple and direct method for the isolation of levulinic acid from the reaction mixture was worked out.

Experimental work : The laboratory work completed *(loc. cit.)* was transferred to pilot plant scale. Initial difficulties encountered during vacuum distillation of the reaction mixture (after removal of HCl - water azeotrope, it became very viscous owing to separation of a large mass of inorganic matter) were partially overcome. The yields of 45 per cent could not be reproduced. Only 30 per cent yield was obtained.

It was thought that yield might improve by using liquid-liquid extraction instead of subjecting the extract to distillation. Benzene, trichloro ethylene, dioxan, commercial hexane, isopropyl ether, ethyl acetate, etc., were tried, but none of these gave better yields.

Preliminary work indicated that if the extract was distilled under vacuum the yield was better. The pilot plant work was done under vacuum but the yield of levulinic acid was observed to be 30 per cent.

Further work: The work will be continued to improve the yield of levulinic acid and also effect elimination of the catalyst.

12. ENTEROVIOFORM

Object: To establish optimum conditions of preparing enterovioform through different routes.

Introduction: Methods of preparing enterovioform are described in literature and it is also being manufactured in India. However, it was observed that its preparation according to the described methods was not always successful.

Experimental work:

(a) *8 - Hydroxy quinoline* (oxine):

Several methods were tried using *o*-nitro and *o*-amino phenol and carrying out the Skraup's synthesis using different moderators. Optimum conditions were established.

(b) *5 - Chloro 8 - hydroxy quinoline:*

Chlorination of oxine with sulphuryl chloride, according to the method described in literature, did not proceed at all. Optimum conditions were then worked out.

(c) *5 - Chloro 7 - iodo - 8-hydroxy quinoline*:

This was obtained by the iodination of the 5 - chloro compound according to the described methods, but minor modifications were necessary to get the U. S. P. product.

It could also be obtained by known methods by direct chlorination - cum - iodination in one step from oxine using iodine trichloride.

Future work: The work will be continued as stated in the objective.

Note: This scheme is being carried out as technical assistance to Messrs. Biological Products Ltd., Bombay.

Essential Oils and Aromatic Chemicals

13. *CYMBOPOGON MARTINI* (PALMAROSA & GINGER GRASS) OILS

Introduction: The schemes under this project envisage the survey, examination and utilisation of essential oils obtained from wild-growing grasses belonging to the family *Cymbopogon martini*. A preliminary report of the survey carried out in Andhra Pradesh, and some parts of Bombay and Madras is under preparation (Annual Report 1957-58 pp. 41-42).

13. 1. Beneficiation of palmarosa oil

Object: To obtain perfumery grade oil of high geraniol content.

Previous work: Concentration of oxygenated compounds by extracting with aqueous solutions of certain salts of organic acids is known. This technique was applied to these essential oils. Rectification of palmarosa oil by steam or vaccum-distillation was carried out earlier (Annual Report 1955, pp. 21-22; 1956-57, pp. 28-29).

Experimental work and results: Aqueous solutions of sodium salicylate as media for hydrotropic beneficiation of geraniol and its esters in palmarosa oil were examined. While geraniol was extracted preferentially by the hydrotrope, the esters were divided almost evenly between the unextracted portion (terpenes) and the hydrotropic solution. With sodium salicylate solutions, terpene-free geraniol could not be obtained, and all the geraniol present in the oil could not be extracted completely. In a typical experiment, 100 ml. of palmarosa oil (total geraniol 57 per cent) were shaken with 400 ml. of 47.4 per cent aqueous solution of sodium salicylate. The unextracted oil (70 ml.) contained 46.9 per cent total geraniol. The aqueous extract on dilution gave 25 ml. of oil having total geraniol content of 88 per cent. In a comparative experiment, the oil was extracted with 55 per cent aqueous alcohol when the up-graded oil of 72.5 per cent total geraniol was obtained. Palmarosa oil with a total geraniol content of 80 per cent and above could not be rectified by this technique on account of complete solubility of the oil in the aqueous solution.

Future work: To improve the beneficiation technique, to investigate other hydrotropic compounds, and to extend the studies on other essential oils.

13. 2. Cultivation of palmarosa grass

Object: To cultivate Ellichpur variety of grass in " moram " soils and to examine the essential oil obtained.

Previous work: Seeds of palmarosa procured from the Forest Department, Madhya Pradesh, were sown separately by broadcast method and in rows (Annual Report 1957-58, p.43).

Experimental work and results : When the average height of grass in both the fields was 5 ft., flowers, leaves and stems were separately collected and steam-distilled. The two oils had the following characteristics respectively :

Yield, 0.466 and 0.22 per cent; density at 28°C., 0.8791 and 0.8775; refractive index at 28°C., 1.4712 and 1.4708; free ester, 10.5 and 5.9 per cent; total ester after acetylation, 93.6 and 95.6 per cent; and total alcohol as geraniol, 91.5 and 94.1 per cent.

It shows promise of obtaining high-geraniol-content palmarosa oil from cultivated varieties of *C. martini*.

Future work : To verify the above results from 1959-60 crops and extend the scope of work in cooperation with the Forest and Agriculture Departments of Andhra Pradesh Government.

14. INDIAN CINNAMON LEAF OIL

Object : To examine the chemical constituents of Indian cinnamon leaf oil and to find new applications of these constituents.

Previous work : Indian cinnamon leaf oil has not been exhaustively studied earlier. Eugenol, its chief constituent has been tried with success as the starting material to manufacture vanillin and 'Lounginin' (see project report on LOUNGININ).

Experimental work : In order to study the cinnamon leaf and bark oils industry, one of the estates in Kerala was visited and discussions were held with local producers and officials of the government. Methods of distillation in practice were examined and samples of various types of oils obtained. Oil obtained from Anjerakandy was selected for detailed examination. After removal of phenols by dilute caustic alkali, the non-phenolic residue was fractionated. Identification of the constituents is now in hand.

Future work : The work as outlined above will be continued.

15. LOUNGININ

Object : Synthesis and pilot plant production of ' Lounginin, ' a substitute for vanillin which is at present imported in India. ' Lounginin ' has twenty times the flavour strength of vanillin. Its

production in India would result in the utilisation of cinnamon leaf oil, an indigenous raw material.

Previous work: Optimum conditions were established for the comparatively inexpensive synthesis involving demethylation with sodium ethoxide in alcoholic solution and a patent application covering this process was filed. Eugenol, the starting material for the synthesis of 'Lounginin,' was obtained from cinnamon leaf oil instead of clove oil which is not readily available.

'Lounginin' was tried in ice-cream and sweet preparations in the cafetaria of the Laboratory, by a local biscuit manufacturer and a restaurant, and was well received. (Annual Report 1957 - 58, pp. 40 - 41).

Experimental work: Cinnamon leaf oil was found to be the best indigenous source for obtaining eugenol. The outer most cinnamon bark shavings were also distilled to get the oil.

Conditions for extraction of eugenol from the oil by caustic soda were standardised, and the eugenol thus obtained was subjected to ethylation (a) after recovery, and (b) as such, in caustic soda solution. The isomerisation step was avoided. Demethylation of ethyleugenol was studied in detail. About 20-25 per cent of a liquid product, whose identity is being established, was simultaneously obtained along with 'Lounginin.'

Having established optimum conditions for different steps on a laboratory scale, the synthesis was carried out on a bench scale producing 500 g. of 'Lounginin' per batch. Various methods for refining crude 'Lounginin' were studied.

One cinnamon estate in Kerala was visited. The Government of Kerala has been requested to take steps to increase the cinnamon plantation.

Future work: Studies on the chemical composition of (i) cinnamon leaf oil, (ii) the liquid product obtained along with 'Lounginin' during its synthesis; and (iii) continuation of pilot plant trials on the production of 'Lounginin.'

16. UTILISATION OF TURPENTINE

The schemes under this project are directed towards utilisation of turpentine *(Pinus longifolia* Roxb.) as a raw material for fine

:hemical industry (see, *inter alia*, Bhushan and Zaheer, *J. Sci. Industr. Res., (India,)* 1953, *12 A*, 287-90; *Chemical Age (India,)* 1955, *6*, 65-69; Annual Report 1956 - 57. pp. 30-31, 1957 - 58, p. 43).

.6. 1. Synthesis of menthol from 3-carene

Object: To prepare menthol and its derivatives.

Previous work: Menthol, a natural product, is obtained chiefly rom aromatic plants of "*Mentha*" group. Several procedures or the synthesis of menthol are also known wherein various start- ng materials have been employed. These syntheses involve a number of steps with progressively decreasing yields of the intermediates.

Attemps were therefore made to prepare menthol in economic yields, using 3-carene as the starting material (Annual Report 1957–58, p. 43).

Experimental work and results: The chlorohydrins obtained from 3–carene were reduced to a liquid having a pronounced peppermint odour. Apparently it was a mixture of various isomers of menthanol. Attempts to isolate the constituents and to prepare solid derivatives for identification having failed, the product was oxidised to the corresponding ketone (2 : 4–dinitorphenyl hydrazone, m. p. 120-122°C.). The ketone was brominated, and the dibromo–derivative was treated with quinoline. The debrominated product was soluble in caustic alkali and gave qualitative tests for thymol. The ketone obtained above was further oxidised to a mixture of acids which are being examined by chromatography and other techniques. The peppermint smelling liquid is also being investigated by gas–liquid chromatography, infra–red spectra and other techniques.

Among other routes to synthesise menthol from 3–carene, which are also being investigated, are the preparation of 3–halo- , 3 : 8–dihalo- , and 3 : 8–dihydroxy–*p*–menthanes as the first intermediates. The cleavage of 3–carene to varying degrees of *para* and *meta* compounds is also being studied.

Future work: The work will be continued as outlined above.

16. 2. Preparation of hydroxylated terpenes

Object: To obtain compounds of potential or known use in perfumery, ore–flotation, surface–coating and textile industries.

Previous work : Various established procedures are known for the preparation of organic compounds having hydroxyl groups. Preparation of such compounds from the constituents of Indian turpentine has been under investigation in the Laboratory for some-time. It has been reported (Annual Report 1957–58, p. 43) that catalytic addition of low molecular weight fatty acids to the olefinic double bond of 3-carene gave low yields of esters, which were obtained as intermediate products for the preparation of corresponding hydroxy- compounds.

Experimental work and conclusions : Mono- and dihalo- terpenes were prepared by halogenation of 3-carene, which were later reacted with sodium salt of a fatty acid. The conditions of reaction favoured dehydrohalogenation resulting in not very encouraging yields of the corresponding esters. Modified procedures are being tried, particularly, to replace the halogen directly by the hydroxyl group. Preliminary experiments are in progress.

Laboratory studies have also been commenced on the production of terpineol and terpinhydrate from Indian turpentine. As a first step, the work of Bhushan *et al.*, *(J. Indian Chem. Soc., Ind. & News Ed.*, 1944, *7*, 62*)* is being repeated to collect data for pilot plant work.

Further work : The work will be carried out as outlined above.

16. 3. Chemical re-examination of Indian turpentine oil (*Pinus longifolia* Roxb.)

Object : To study the constituents of Indian terpentine oil.

Previous work : Indian turpentine was exhaustively examined by Simonsen and others around 1920 with the then available techniques. Re-examination is being attempted by modern techniques to isolate constituents which might have escaped detection by the earlier workers. Turpentine oil was repeatedly fractionated and the major constituents identified by known methods. Physical characteristics of each fraction were also determined.

Experimental work : Each fraction was examined by gas-liquid chromatography. Thirteen constituents have been detected so far. Infra-red spectra of selected fractions were also obtained (by the courtesy of the Physics Department, Osmania University, Hyderabad).

Future work : Suspected mixtures will be refractionated on a microdistillation column. Spectroscopic work will be completed and the identity of new constituents established.

16. 4. Isomerisation of 3-carene

Object : To obtain products which may have direct application in industry or may serve as starting materials for other useful industrial products.

Previous work : Bhushan, Verghese and others have studied catalytic isomerisation of 3-carene in vapour and liquid phases. The reactions carried out by them employed conditions which were conducive to obtaining the various menthadienes, chiefly α-terpinene, *m*- and *p*-cymenes, menthane and some polymers.

Experimental work and results : Preliminary experiments were carried out using milder catalysts and the products are being investigated.

Future work : Work will be continued as indicated above.

17. PESTICIDES

17. 1. Production of 'Citicide' (Chlorinated turpentine)

Object : To produce effective and relatively inexpensive insecticide from turpentine oil.

Previous work : 'Citicide,' a potent insecticide, was obtained by exhaustive chlorination of turpentine oil. Pilot plant production of 'Citicide' was also carried out (Annual Report 1957-58, pp. 44-45).

Experimental work and conclusions :

(a) *Pilot plant production* : Pilot plant experiments concluded earlier, were evaluated and project costs and other details were published (Bharat Bhushan *et al.*, *Res. & Ind.*, 1958, *3*, 267). The process has since been handed over to the National Research Development Corporation of India for negotiating its commercial exploitation.

Chlorination was carried out continuously in the pilot plant using about 30 kg. of turpentine per batch. Ten batches were run to a chlorine content of 60–63 per cent. A large number of samples were supplied to interested parties at home and abroad.

Changes in physical characteristics of the product during the course of chlorination were compared with the chlorine content, with a view to finding a means of quick and easy control during commercial production. A linear relationship between the chlorine content and the refractive index, viscosity or density being not established spectroscopic methods are being investigated.

(b) *Storage and packing*: In addition to confirming the earlier results that the stabilised 'Citicide' could be stored in metal containers for long periods, it was found that polythene containers could be used for storage and packing of this product.

(c) *Production of 'Citicide' of higher toxicity*: The procedure for the initial chlorination of turpentine is being modified for obtaining 'Citicide' of higher chlorine content and hence, possibly, with higher toxicity. The work on this aspect will be taken up.

(d) *Samples for entomological evaluation*: Samples for evaluation of toxicity of 'Citicide' were sent to Central Food and Technological Research Institute, Mysore, and Defence Research Laboratory, Kanpur, for laboratory tests, and to Messrs. Tata-Fison and Pest Control Ltd., Bombay, for field tests. The test reports are awaited.

Future work: Work will be continued as outlined above. In addition, preparation of other Toxaphene-type compounds will be undertaken.

17. 2. Preparation of thiocyanoacetates of terpenes

Object: To prepare toxic compounds having pronounced knock-down action on insects.

Previous work: Carane-3-thiocyanoacetate prepared in 50 per cent yield was highly toxic to houseflies but was devoid of the expected knock-down properties (Annual Report 1957-58, pp. 47-48).

Experimental work: The three-step procedure to obtain carane-thiocyanoacetate *via* corresponding chloro-acetate gave very low yields of the intermediate product. The procedure was therefore modified and 3-hydroxy carene was obtained in good yield.

Future work: The work will be continued on the above lines and will be extended to the preparation of Thanite by a new route.

17. 3. Toxicity studies on 'Citicide'

Object: To study the toxicity of chlorinated turpentine on various insects, with reference to the relative importance of factors like temperature, formulation, etc.

Previous work: Houseflies were exposed to different temperatures after treating them topically with chlorinated turpentine. The mortality after 2 hours at 33°, 35° and 37°C was in males 58, 90 and 100 per cent and in females 30, 44 and 70 per cent respectively. When houseflies were exposed in a cage to the surface of glass panels sprayed with 4 per cent chlorinated turpentine, it took longer to produce hundred per cent mortality in females than in males.

Experimental work and conclusions: Studies on the effect of different temperatures on the toxicity of chlorinated turpentine were continued. Preliminary experiments were carried out by exposing houseflies to 25°C. after treating them topically with 5 per cent chlorinated turpentine in acetone. The percentage of mortality after 2 hours in males and females was 36 and 30 per cent respectively. Work on the effect of 'Citicide' at lower temperatures is in progress.

Future work: (i) To study the toxicity of chlorinated turpentine at lower temperatures (25°-15°C.); (ii) a comparative study of toxicity of six well-known insecticides at different temperatures using topical and surface contact methods.

Entomology

18. ENTOMOLOGICAL STUDIES ON INSECTICIDES

18. 1. Mechanism of synergism

Object: Considerable work has appeared in literature on synergism between insecticides. However, the mechanism involved has not been clearly understood. Such knowledge will greatly help in making effective combinations of insecticides.

Previous work: The site and mode of action of sesamin and Sesoxane was studied using the isolated cockroach-heart technique described by Naidu (*Bull. Entomol. Res.* 1955, *46*, 205). Sesamin

did not modify the action of nicotine and allethrin. Eserine potentiated the stimulant effect of sesamin, while atropine inhibited it. It was presumed that sesamin did act mainly at the neuro-muscular region. Sesoxane stimulated the heart-beat of male and female cockroaches. The stimulant action of Sesoxane was suppressed by atropine.

Experimental work and conclusions: To study the site and mode of action of Sesoxane further experiments were carried out on cockroaches using isolated heart technique. Sesoxane at concentrations of 6×10^{-5} to 10^{-5} caused an immediate rise in the frequency of the heart-beat in males followed by a decline whereas, in females, the degree of rise in the frequency was comparatively less for the same concentration. Sesoxane potentiated the action of pyrethrum. The results obtained with higher concentrations of pyrethrum were however inconclusive. Previous treatment with Sesoxane (10^{-5}) interfered with the stimulant action of nicotine; this was also true when the treatment was reversed. Low concentration of Sesoxane did not interfere with the stimulant action of nicotine and this was also true when the treatment was reversed. After prolonged action with nicotine (5×10^{-5}) for 3 hours, Sesoxane (6×10^{-5}) caused little or no increase in the frequency of the heart-beat. It would be concluded that Sesoxane did primarily act at the neuro-muscular junction and partly on the cardiac ganglia and that acetyl-choline must be involved in its action.

Future work : It is proposed to work on piperonylbutoxide, another powerful synergist for pyrethrum, and other suspected synergists.

18. 2. Sex differences in insecticide tolerance

Object : To study the causes for the sex difference in resistance to insecticides.

Introduction and previous work : Greater tolerance of female insects to insecticides compared to males of the same species is well known. Some workers believe that differences in fat content could be of major importance. Isolated cockroach-heart technique was selected for this investigation, as it did not involve body weight and fat. Pyrethrum caused an increase in the frequency of the heart-beat followed by a decline in adults and nymphs of both sexes. In females,

a delayed decline was caused which was less than that produced by the same concentration of pyrethrum tried on males. Such variation was also found true with nicotine.

Experimental work and conclusions: Experiments were carried out on male and female isolated cockroach-heart preparations using different concentrations of rotenone. It is known that the time-lag between the administration and appearance of the toxic effect of rotenone varies with the concentration. The time-lag in males between the administration and appearance of its effect at a concentration of 2×10^{-6} was 15 - 20 min. in males and females respectively. At lower concentrations (10^{-8}), the time-lag varied considerably between two individuals of the same sex and different sexes. It may be attributed that the discrepancy in results is due to improper dispersion of the insecticide in solutions. It is concluded that more tolerance of female insect to an insecticide is independent of the lipid content of an insect.

Future work: Differential sex susceptibility of insects to insecticides may be due to (i) the physiological differences at the cellular level in the insects, and/or (ii) sex hormones. These two factors will be studied.

18. 3. Physiological action of insecticides at different temperatures

Object: There are two general trends on the effect of temperature on the toxicity of insecticides: (i) increase in toxicity at lower temperatures and (ii) increase in toxicity at higher temperatures. It is intended to study the cause of variations in toxicity at different temperatures and the mechanism involved.

Previous work: Considerable work has been done on the effect of temperature on the toxicity of different insecticides. However, not much attention has been paid to understand the physiological implications involved. Whitcomb (1934) (*Bull. Mass. Agric, Exp. Sta.* No. 305, p. 36) found that the effectiveness of some insecticides varied directly with the increase in temperature and in others it varied inversely. Poffer and Gillham (*Ann. Appl. Biol.*, 1941, *30*, 259) showed that cooler conditions after spraying increased the toxicity of pyrethrum and DDT.

Experimental work and conclusions: To Study this mechanism, isolated cockroach-heart technique was used. Male and female isolated cockroach-heart preparations were subjected to different temperatures (20°-40°C). As the temperature was raised, the frequency of heart-beat also increased. At all temperatures except 20°C. the frequency of the heart-beat of male cockroach was considerably more than that in females.

Acetylcholine and eserine increased the frequency of the heart-beat at all temperatures tested. Addition of atropine inhibited considerably the increased frequency of the heart-beat at higher temperatures.

Future work: To study further the effect of cholinongic and adrenongic drugs at different temperature on coackroch - heart.

18. 4. DDT-resistant houseflies

Object: To breed highly DDT - resistant houseflies as a preliminary to devising insecticides and synergists potent against them.

Previous work: The method followed to develop resistance in houseflies against DDT was to continuously breed the survivals of DDT-treated flies in various generations. Twenty-fourth generation of DDT-treated flies exhibited some resistance to DDT, but this was not noticeable in the next generation.

Experimental work and conclusions: A sub-lethal dose of DDT was applied to houseflies topically. At present thirty-third generation of DDT-treated houseflies are under culture, and they appear to have acquired some resistance to DDT.

In addition to the topical method a separate batch of houseflies was sprayed with DDT and a sub-lethal dose of DDT was also mixed in the housefly larval media. At present a twelfth generation of houseflies thus treated is under culture.

18. 5. Effect to X-rays on the biological and biochemical properties of the spermatozoa of insects

Object: There has been very little work done on the biological properties of spermatozoa of insects *in vitro*. The primary

object is to determine the effect of X-rays on spermatozoa *in vitro* as a homogeneous suspension.

Previous work: Considerable work on the morphology and physiology of mammalian spermatozoa has been done and this has been reviewed by Bishop and Austin (*Endeavour*, 1957, *16*, 137 - 50). There has been some work on the structure and formation of spermatozoa of insects and their migration in the female tract. But little work has been done on irradiated homogeneous suspension of spermatozoa.

Experimental work and conclusions: Longivity of the spermatozoa of the cockroach *in vitro* was studied in four different types of media. About 50 per cent were alive after 4 hours in the media containing phosphate buffer + 1 per cent fructose. No sign of life was seen in other media.

A detailed examination of the male and female reproductive systems of the cockroach was made by dissecting and staining. Morphological study of the spermatozoa from different regions of the male and female reproductive system was also made by staining the spermatozoa.

Future work: Further study on (i) the longivity of the spermatozoa of cockroach in different media and temperature, (ii) path followed by the spermatozoa while mating in male and female cockroach, and (iii) artificial insemination in cockroaches.

18. 6. Toxicity studies on 'Citicide'

(Please see under PESTICIDES).

Biochemistry

19. ITACONIC ACID

Mechanism of itaconic acid formation by *Aspergillus terreus*

Object: To determine the mechanism of formation of itaconic acid from sucrose or glucose by *Aspergillus terreus*.

Previous work: Under suitable conditions, with cane sugar or glucose as substrate, *A. terreus* converts as much as 50 per cent of

the sugar to itaconic acid. On the basis of the nature of labelling of itaconic acid obtained from C - 14 labelled glucose, acetate and succinate, Bentley and Thiessen (*J. Biol. Chem.*, 1957, *226*, 689) concluded that normal Kreb's cycle operates in this organism and that the immediate precursors of itaconic acid are *cis*-aconitic acid and citric acid, the latter being obtained in the usual way from oxaloacetic acid and *acetyl-Co* A.

A study of the mechanism of itaconic acid formation was begun in this laboratory before the publication of Bentley and Thiessen's observations. The effect of fluoride, iodoacetate, azide and cyanide on itaconic acid formation and sugar utilisation has been reported earlier. The effect of supplementing the sugar medium with 1 - 3 per cent of citric, *trans*-aconitic, malic, succinic or fumaric acid (at pH 3) on itaconic acid formation has also been described. The results of some preliminary experiments using acetic acid (at pH 3 or 5) or unneutralised sodium acetate, in addition to sugar, were also reported (Annual Report 1957–58, p.52).

Experimental work and conclusions: Experiments with Kreb's cycle intermediate were extended and confirmed. Citric, malic, succinic and fumaric acids were poor substrates for itaconic acid formation even at a concentration of 8 per cent. Addition of 1 per cent sugar had little effect. The role of pyruvate and acetate in itaconic acid formation was studied and the results obtained are briefly described below:

(a) maximum inhibition of itaconic acid formation from sugar, by sodium acetate, is obtained at 1.5 - 2 per cent concentration of the latter. The utilisation of sugar remained more or less unaltered in the presence of acetate, while mycelial growth was stimulated;

(b) at a substrate (acetate or sugar) concentration of 4, 8 and 12 per cent respectively, acetate gave 58, 65 and 20 per cent of the yields of itaconic acid obtained with the corresponding concentrations of sugar;

(c) 3 per cent pyruvate completely reversed the inhibition of itaconic acid formation from sugar by fluoride; the sugar consumption is also fully restored;

(d) 3 per cent pyruvate also reversed the inhibition of itaconic acid formation by iodoacetate, although to a much lesser

degree than that obtained in the case of fluoride; at fourteen days 60 per cent of sugar consumption in control was restored ;

(e) 1 - 3 per cent pyruvate had, usually, no effect (in some cases a slight stimulation of itaconic acid formation was observed) on the conversion of 15 per cent sugar to itaconic acid, but increased the mat weight by 25 - 50 per cent of the control ;

(f) pyruvate in 1 - 3 per cent concentration was a slightly better substrate than sugar (in spite of pH rising to more than 10 from 3, at the end of the fermentation period of 7 - 14 days) :

(g) acetate did not reverse the inhibition of itaconic acid formation from sugar by fluoride and iodoacetate, although a partial restoration of sugar consumption was obtained ;

The above observations are in line with the conclusion arrived at earlier that acetate (or *acetyl-Co* A) was not an intermediate in the conversion of sugar to itaconic acid.

Future work : To try pyruvate as a substitute for itaconic acid formation, keeping the pH of the medium between 3 - 8, and to study the effect of adding enzymes and co-enzymes involved in the conversion of pyruvate to *acetyl-Co* A. Positive evidence in favour of direct conversion of pyruvate to itaconic acid will be sought.

20. CALCIUM GLUCONATE

Microbiological production of calcium gluconate

Object : To study and work out the details of a process for the microbiological production of calcium gluconate on an industrial scale.

Previous work : The details of the method worked out in this laboratory for the production of calcium gluconate from glucose by *Aspergillus niger* NRRL 67, have been described earlier (Annual Report 1957-58, p. 50).

Experimental work and conclusions : The method described earlier was extended to liquid glucose, since liquid glucose would be a much cheaper raw material than glucose.

An average yield of approximately 50-60 per cent on the basis of the initial reducing sugar content of liquid glucose has been obtained in a series of experiments.

A complete analysis of a sample of calcium gluconate prepared from glucose earlier in this laboratory was carried out. The product conformed to the B. P. standards.

Future work: To carry out a few more experiments using liquid glucose with a view to further standardise the conditions, and determine the optimum number of runs which could be made with the same batch of the mould. Preparation of ferrous gluconate by suitable modification of the process will also be attempted.

21. CITRIC ACID

Microbiological production of citric acid

Object: To develop a suitable process which could be used for the industrial production of citric acid from cane sugar source (like molasses) by *Aspergillus niger*.

Previous work: Details of the investigations leading to the development of a process giving good yields of citric acid from cane sugar on laboratory scale have been described earlier (Annual Report 1956-57, p.33).

Experimental work and conclusions: Using phosphoric acid to adjust the pH of the culture medium a yield as much as 50 per cent on the basis of sugar taken, could be obtained in the laboratory. Studies using methanol and/or zinc sulphate are in progress. When glucose was used as the substrate in place of sucrose, results similar to those mentioned above were obtained. Attempts to produce citric acid from acid-hydrolysed starch and *mahua* flowers did not meet with much success. In experiments with clarified molasses as the sugar source, 50 - 55 per cent yields of citric acid (based on the initial sugar content of the fermentation medium) were obtained.

Conditions were worked out and standardised for the production of citric acid from cane sugar in 40 - 45 per cent yields on a semi-large scale. Fermentation carried out in enamel, aluminium or stainless steel trays gave low yields of citric acid. Enamel or other

trays lined with polythene film gave results as good as those obtained with glass trays.

Investigations were also carried out in selecting a proper strain of *A. niger* for the production of citric acid by submerged fermentation. About twenty different strains were tested in 250 ml. shake flasks containing sucrose medium. Except for B_8 and NRRL 372, all the other strains were poor acid producers. Thus, strains found active for acid production by surface culture were unsuitable in submerged fermentation. Citric acid production in shake flasks was found to be highly sensitive to changes in the incubation temperature and pH of the culture medium.

Future work : (i) To improve yields of citric acid from cane sugar on semi-large scale, (ii) to develop a process utilising molasses which will be capable of industrial exploitation; and (iii) to study the production of citric acid by submerged fermentation.

22. CELL CONCENTRATION EFFECT

Effect of cell concentration on the biochemical properties of individual cells in cell suspensions

Object : To study the dependence of the rates of various metabolic reactions in individual cells, on the concentration of cells in the cell suspension.

Introduction and previous work : From some earlier observations it appeared that the higher the concentration of cells in a medium, the lower was the metabolic rate per cell. This phenomenon has been termed as 'Cell concentration effect' by workers in this laboratory. It is possible that this effect may have an important role to play in regulating growth. The aim of this investigation is to determine the range of applicability and the biological significance, if any, of the above effect, by using different types of cell suspensions like bacteria, spermatozoa, blood cells, tumour cells, etc., and studying the rate of their metabolic reactions at different cell concentrations. No systematic investigation seems to have been carried out along these lines earlier, although there is some indirect evidence available in the literature suggesting that this effect is generally obtained in normal cells.

Experimental work and conclusions: The growth of *Pseudomonas fluorescens* in a peptone-beef extract medium has been studied in detail. If the cell concentration effect were to operate in a growing bacterial culture, the rate of synthesis of bacterial protoplasm would fall with increasing cell population. Although this could be inferred by recorded observations in the literature, this does not seem to have been pointed out or adequately discussed, nor its biological implications understood. It has now been found that the time (T) taken for the bacterial protoplasm to double itself in mass increases continuously in a growing culture, even during the log phase of growth. It was found that T was independent of (i) the age of the parent cultures used for inoculation, 1, 3, 5, 7, 9 and 20 hr. old cultures having been tried, and (ii) the size of the initial inoculum. T, however, depended on the strength of the medium used. For most of the experiments a medium containing 48 g/l of peptone and beef extract was used. At this concentration, nutrient did not appear to be the factor limiting growth. The cultures were shaken when they grew as a uniform suspension. Mass was measured turbidimetrically. Eight to ten hours were required for the maximum stationary growth stage to be reached. The above observations are in accord with what one would expect on the basis of cell concentration effect.

Future work : It is possible that the cessation of growth after the maximum stationary phase has been reached, may be due to the cell concentration effect in those cases where nutrient and/or accumulation of toxic products are not the limiting factor. It is proposed to gain more information on the above point in future. This work will then be extended to other properties, like respiration, protein, nucleic acid and fat metabolism, enzyme activity, etc., of bacteria, blood cells, spermatozoa and ascites tumour cells.

23. PROTEIN AND NUCLEIC ACID BIOSYNTHESIS AND METABOLISM

Nucleic acid, protein, free nucleotide and free amino acids of spermatozoa

Object: To study the nucleic acid, proteins, free nucleotides and free amino acids of various kinds of spermatozoa.

Introduction and previous work: Since spermatozoan is considered to be a specially interesting cell for studying protein and

nucleic acid metabolism on account of the reported absence of RNA from this cell, a very low free amino acid pool and a high content of free nucleotides, a detailed analysis of the proteins, nucleic acid, free nucleotide and free amino acids of spermatozoa and seminal plasma was undertaken. Proteins of spermatozoa and seminal plasma have been examined in some detail by previous workers, and RNA has been shown to be absent from spermatozoa of about half a dozen species. Free nucleotides and free amino acids of semen have, however, received little attention in the past (Mann, *"Biochemistry of Semen"*, Cambridge University Press, 195; Bhargava, *Nature*, 1957, *179*, 112; Bhargava, Bishop and Work, *Biochemical Journal*, 1959, in press).

Experimental work and conclusions: Spermatozoa of bull (several breeds), buffalo bull and goat have been shown to contain similar amounts (0. 1 - 0.2 x 10^{-9} mg/cell) of total free nucleotides. From the seminal plasma of these animals a protein was isolated which was not denatured, although precipitated, by 5 per cent trichloroacetic acid (TCA), which was soluble in dilute salt solutions but insoluble in water, and which was mostly dialysable suggesting that it was of low molecular weight. This protein, which is absent from spermatozoa, may represent as much as 50 per cent of the dry weight of seminal plasma and 90 per cent of the total TCA-precipitable proteins of the plasma.

Future work: The above work will be further extended. Attempts will be made to characterise the protein fraction described, and to determine its biological function. This analytical work will be followed by a study of the incorporation of suitable precursors in the protein and nucleic acid of spermatozoa.

Hand-made Paper and Cellulose

24. HAND-MADE PAPER

Object: To carry out pilot plant research to study manufacture of special quality papers like bond, document, drawing and filter papers, mainly imported at present. This is with a view to develop an essentially hand-based small-scale industry in India.

Introduction and previous work: The project was started in 1953. Various operations involved in production of high-quality papers by essentially hand-made methods have been systematically studied with

the aim of improving product quality, increasing production and reducing working costs. The saleable products obtained during the investigation were marketed.

A new type of paper lifting vat, the "Centro-vat", which has a two-fold advantage of less physical exertion and improved production was designed, fabricated and put into operation. Strength of paper was improved by using a larger proportion of new rags by replacing caustic soda with soda ash for digestion, and by altering the freeness of the pulp.

Work during the year:

High bursting strength paper: Work on paper according to the specifications of the Ministry of Defence, Government of India, was taken up. Linters, rags and bamboo were tried. A pulp composition was worked out and the paper sent for tests which met with all the specifications except that it did not possess enough stretch. Further work is in progress. This quality of paper is at present imported.

Filter paper: Studies were made on preparing filter paper from new white rags. Different digestion conditions were tried and pulps of different degrees of freeness obtained. Besides laboratory experiments a pilot plant batch was also run. The filter sheets tested for filterability and retention, and analysed for ash, proved satisfactory.

Further work is in progress using bleached linters as starting material. Preliminary experiments have yielded a very satisfactory quality of filter paper for qualitative work. A pilot plant batch was also run.

Hand-made paper: Improvements in quality in respect of uniformity, whiteness and removal of specks have been effected by better control over beating, modifications in lifting methods and changes in sizing and other additives. In uniformity and whiteness, the paper has come to be almost as good as imported hand-made paper. The sources of specks are being traced and systematically analysed so as to make a paper completely free from specks.

Drawing paper: Experiments carried out on coarse grain drawing paper for artists were successful.

Card paper, ledger paper and document paper : A number of small batches of these papers were produced.

Fuel

25. LOW-TEMPERATURE CARBONISATION OF NON-CAKING INDIAN COALS

25. 1. Pilot plant production of smokeless domestic fuel

Object : To produce a smokeless domestic coke from locally available high-ash, non-caking coals and market it, with a view to (i) familiarise the population of the region with the advantages of using domestic coke; (ii) gradually displace domestic fuels like charcoal and firewood with semi-coke; and (iii) create a favourable technical and socio-economic basis for the establishment of a large-scale low-temperature carbonisation (l. t. c.) industry in the region.

Previous work : A Lurgi-spuelgas type of l. t. c. pilot plant, with a capacity of 25 tons of coal/day, has been in regular operation since 1954. Four types of non-caking and weakly caking coals from different coalfields in India were successfully carbonised (Annual Report, 1957-58, p. 59).

Work during the year : Production of the smokeless domestic coke 'KOLSIT' by low-temperature carbonisation of coal at 660°C. was continued. During the year, about 4,762 tons of Kothagudem coal were carbonised and 2,489 tons 'KOLSIT' (over 18 mm. size) were marketed, compared to 3,400 and 1,800 tons respectively during 1957-58.

The plant was maintained in working order. The two conveyors of the plant were provided with sheet metal covering. A new track balance was installed and a shed provided for it. The main aluminium chimney of the predryer was replaced by a new one. Repairs were done to the electric hoist; trolley tracks were re-modelled and new turn-tables fitted.

Future work : The above work will be continued.

25. 2. Tests on combustion of semi-coke

Object : (i) To determine the optimum conditions for the efficient combustion of various sizes of semi-coke; (ii) to design ovens

for efficient and controlled combustion of semi-coke of size 18-25 mm. to meet domestic and other requirements of consumers of different economic strata.

Previous work : Preliminary tests showed that 14.5 min. were required to ignite a bed of semi-coke of size 18-25 mm., weighing 3.1 kg. and spread over a grate of 0.093 sq.m. area and that a maximum temperature of 570°C. was reached after 45 min.

Experimental work and conclusions : Combustion tests were continued, using in each test, 6.2 kg. of semi-coke of size 18-25 mm., spread over a grate of 0.093 sq.m. total area, of which 55 per cent was open. The height of the fuel bed was 90-100 mm. over the grate. Temperatures were measured at regular intervals of time at 9 points of the oven, one of which was just on the grate, another 38-40 mm. above fuel bed (corresponding to the centre of the combustion chamber) and the others distributed on the cover plate and chimney. The influence of primary air and natural draught on the rate of rise of temperature was studied. Plots of temperature *vs.* time with reference to temperatures at the centre of the combustion chamber and on the grate showed that there was at first a pre-ignition stage, followed by combustion and cooling stages. When the temperature on the grate reached 600-700°C., there was a sharp increase in the rate of rise in temperature above the fuel bed. Complete closure of primary air portholes and choking of the chimney draught before or at the attainment of maximum temperature above the fuel bed, helped in prolonging the total period of combustion. A maximum temperature of about 900°C. was attained at the centre of the combustion chamber in one hour after igniting of the fuel bed.

Future work : Optimum conditions will be worked out to maintain a temperature of 400°C. and above for the longest duration. Combustion characteristics of semi-coke of 12-18 mm. size will also be studied.

25. 3. Market survey of the products of low-temperature carbonisation of coal

Object: (i) To estimate the potential consumer demand in the region for the various products of low-temperature carbonisation of coal and secondary products therefrom with a view to establish the

economic feasibility of a large-scale industry; (ii) to assess the consumer reaction to 'K O L S I T' with a view to improve its properties.

Previous work : Replies received to the questionnaires on potential demand for 'Kolsit' were analysed statistically. A field survey based on personal enquiry was considered necessary. A statistical sample for the purpose was prepaped (Annual Report 1957-58, p. 60).

Report of work : The survey in the city of Secunderabad was completed. The spheres of utilisation selected for the purpose of survey were residential houses for domestic consumption, and hotels, restaurants and small scale industries. The consumption was estimated on the basis of figures supplied by dealers in fuels.

A list of fuel depots selling different types of fuel, supplied through the courtesy of Municipal Corporation, Secunderabad, provided the frame-work of the survey. Stratified random sampling technique was followed in selecting the sample, and data collected by personal interviews.

Annual consumption of diferent types of fuel was estimated to be as follows :—

Domestic sphere	8,760 tons
Hotels, restaurants and small scale industries	14,256 tons

The prices of different types of fuel varied in different seasons as follows :

	Price per ton Rs.
Coal	47 — 48
Charcoal	144 — 168
Firewood	51 — 56
Coke	70 — 85

Future work : Based on experience in Secunderabad City, the plan for Survey for Hyderabad City has been prepared. The necessary basic data have been collected and field work commenced. This will be completed.

26. UTILISATION OF THE PRODUCTS OF LOW-TEMPERATURE CARBONISATION OF COAL

26. 1. Preparation of pitch and road tar from low-temperature tar

Object : Preparation of different grades of pitch and of different types of road tar, conforming to Indian Standards Specifications, from low-temperature tar.

Previous work : For the production of different grades of pitch, the heavy tar fraction was used as the starting material and the technique of air - blowing was used. Pitch suitable for blending with tar oils for the preparation of road tar could be obtained in 85 - 90 per cent yield with an air - flow rate of 0.5 to 1 cft./lb. hr. for 3 to 4 hours at 300° to 320° C. Stirring during air - blowing operation reduced up to 50 per cent the time required to get a pitch of a given softening point with only a slight decrease in the yield of pitch.

Experimental work and conclusions : Further experiments were conducted to study the effect of stirring during air - blowing in order to determine the optimum conditions of operation to get grades of pitch suitable as base for road tar preparation.

The effect of the presence of catalysts during air - blowing to get a pitch of a given softening point was not reduced when phosphorus pentoxide and manganese naphthenate were used as catalysts, but when ferric chloride was used the duration was reduced by about 30 per cent. Hard pitches prepared by catalytic air blowing were found to exude small quantities of oil on the surface indicating immiscibility with oil (non - homogeneity).

Bulk quantities of different grades of air - blown pitches and blending component, viz., fractions of heavy tar and of neutral oil from light tar, were prepared for blending experiments. Of the blending agents tried, only the heavy tar and the +300°C. fraction from neutral oil were found to be compatible with air - blown pitches.

Eleven samples of road tar closely conforming to the RT_4 grade of ISS (1951) were prepared from low - temperature tar and are being tested at the Central Road Research Institute, New Delhi.

Future work : (i) Further laboratory work will be taken up, if necessary, after a test report on the above samples is received from the Central Road Research Institute. (ii) Different grades of road tars will be prepared on a pilot plant scale and subjected to field tests.

26. 2. Preparation of creosote for timber preservation from low-temperature tar

Object : To prepare different grades of creosote suitable for timber preservation from low - temperature tar.

Previous work : Creosotes prepared from low - temperature tar have good anti - termite and fungicidal properties, but their specific gravity is lower than the requirement of the Indian Standards Specification which is based on creosotes prepared from high-temperature tar.

The light tar (170° - 360° C.) from low-temperature carbonisation of coal was air - blown at 100° - 200° C. for 30 min. to 2 hours at an air rate of 0.25 cft./lb.hr. Although the specific gravity of the product increased from 0.9870 to the desired figure of 1.0315, the benzene insoluble content was more than 3 per cent and the residue above 315° C. sticky, both of which were contrary to the specifications. Distillation of tar with and without air - blowing were tried (Annual Report 1957 - 58, p. 62).

Experimental work and conclusions : Repeated tests showed that distillation of light tar with simultaneous air blowing gave a satisfactory creosote conforming to Type I creosote of ISS No. 288 except for specific gravity. Ninety four gallons of heavy creosote ISS Type I and four gallons of ISS Type II were prepared by the above methods and sent to the Forest Research Institute, Dehra Dun, in June 1958, for mycological and accelerated field tests. Work on this problem has been suspended pending receipt of a test report on these samples from the Forest Research Institute.

26. 3. Recovery of motor spirit from low-temperature carbonisation gases

Object : To recover and examine the crude motor spirit from the mixed gases given out by the Lurgi - Spuelgas low - temperature carbonisation pilot plant.

Previous work: Based on the laboratory tests, an active carbon absorption unit with accessories was designed, fabricated and installed for the recovery of one gallon of crude motor spirit per day from the circulation gases produced in the low-temperature carbonisation pilot plant. In several tests conducted on this unit the crude motor spirit could not be obtained uncontaminated since the oil droplets entrained in the gases fouled the active carbon bed. Modification of the oil-separation unit to one of packed baffle-type, reduced the fouling but could not eliminate it entirely (Annual Report 1957 - 58, p. 63).

Experimental work and conclusions:

(i) The low-temperature carbonisation gases were cooled to 5°-10°C. in a pipe-cooler where most of the entrained oil could be separated. Tar fog, however, persisted and continued to foul the active carbon bed.

(ii) Distillation and chemical examination of crude motor spirit recovered from low-temperature carbonisation gases has been undertaken. The crude product was acid-refined using the S. T. P. T. C. method. Loss on acid-refining was 20 per cent, indicating a higher olefinic content than that occuring normally in coke oven benzol. The colour of the product was pale yellow but the specific gravity was high.

Future work: Work on recovery will be continued using wash oil and low-temperature methods and chemical examination will be continued.

26. 4. Recovery and fractionation of tar acid from tar oils and ammoniacal liquor

Object: (i) To develop methods for the extraction of tar acids from tar oils and ammoniacal liquor obtained during low-temperature carbonisation of coal; (ii) to fractionate the tar acids into industrially useful cuts; and (iii) to study their utilisation.

Previous work:

Tar acids from light tar: Crude tar acids extracted by alkali from low-temperature tar oil were dehydrated and depitched under

reduced pressure and then fractionated into 5 cuts up to 143°C/ 53-60 mm. Three samples comprising (i) a fraction rich in cresols and xylenols boiling mainly at 188° - 213° C/760 mm., (ii) depitched tar acids, and (iii) tar acid residue, were tested at the Central Leather Research Institute, Madras, for their suitability for the preparation of syntans. The fraction rich in cresols and xylenols was found satisfactory (Annual Report 1957-58, p. 64).

Experimental work and conclusions :

(i) Experimental runs were carried out on the alkali extraction of tar acids from low-temperature tar oil distillates using the 500 litres/hour capacity Luwesta Centrifugal extractor. The oil (170° - 230° C.) to alkali (10 per cent) ratio was 1 : 1.5. It was found that whereas the extraction efficiency was 96-97 per cent, there was undesirable sludge formation between the extraction plates of the unit, probably because a stored tar oil distillate was used for extraction. Laboratory tests also showed that a stored oil when treated with alkali formed sediments whereas the fresh distillate did not.

(ii) In order to obtain design data on continuous tar acid extraction units, a detailed investigation has been started on the continuous extraction of tar acids from tar oil distillates using alkali and other extraction media.

Tar acids from ammoniacal liquor: A complete pilot plant for extraction of tar acids from 800 gal / day of liquor is under fabrication. Further work will be taken up after installation of the plant.

Future work : The above work will be continued.

26. 5. Low pressure hydrogenation of low-temperature tar

Object : (i) To investigate methods of preparation of diesel oil, kerosene and lubricants from low-temperature tar by catalytic hydrogenation under low and medium pressures, and (ii) to develop suitable catalysts for this purpose.

Introduction : Low pressure hydrogenation is one of the fast developing newer techniques of utilisation of low-temperature tar. Although commercial size plants of this type do not yet appear to

have been established, the process offers great promise because, it has the advantage of lower capital and operating costs over the conventional high pressure hydrogenation process. This work is of great importance in view of the fact that the large quantities of low-temperature tar produced from the contemplated low-temperature carbonisation plants could be more economically processed into diesel fuel and kerosene for which there is already a shortage in this country.

Experimental work : Preli minary experiments on hydrogenation of a low-temperature tar oil fraction boiling between 225° - 290° C. were conducted in a rocking type Parr hydrogenation apparatus of 50 ml. capacity at a temperature of 360° C. and initial hydrogen pressure of 1300 p.s.i. using 0.5 per cent molybdenum oxide-on-alumina catalyst. The liquid and gaseous products obtained by hydrogenation with or without the catalyst were analysed.

Future work : (i) Continuation of studies on batch and continuous hydrogenation of low - temperature tar and its components such as tar acids, neutral oils, etc., using different catalysts based on molybdenum, tungsten, nickel, etc., with suitable promoters; (ii) study of the kinetics and mechanism of low pressure hydrogenation.

26. 6. Cracking of low-temperature tar

Object : To study the application of thermal and catalytic cracking processes to low - temperature tar for the production of: (i) liquid fuels and products rich in aromatics (C_6–C_{10} boiling range); (ii) fuel gas of high calorific value; and (iii) olefinic gases such as ethylene, propylene, etc., for the synthesis of chemicals.

Previous work : Much of the work done abroad on cracking of low-temperature tar is covered by patents. However, in Germany, commercial scale plants are in operation producing liquid fuels, coke and olefin - rich gases. This problem is of great interest to India in view of the fact that a variety of products such as liquid fuels, aromatics, fuel gases and olefinic gases could be obtained by cracking low - temperature tar that will be produced from the contemplated low - temperature carbonisation plants. The process would not only meet the country's needs for various kinds of fuels but also provide raw materials for a synthetic organic chemical industry.

Experimental work : A bench - scale apparatus consisting of a constant feeding device for the raw oil, reactor and furnace, condensing system and gas holder with constant pressure arrangement was set up. Standardisation of conditions of operation of this unit is in progress.

Fractions of low - temperature tar to be used for the cracking experiments were analysed for hydrocarbon types.

Future work : (i) Studies on thermal cracking of tar fractions and the resulting products will be carried out; (ii) studies on catalytic cracking will be conducted using silica - alumina, silica - magnesia, activated clay and other catalysts.

26. 7. Studies on distillation of low-temperature tar

Object : (i) To study the distillation characteristics of low - temperature tar with a view to provide data for design of distillation and fractionating equipment for processing of low - temperature tar on commercial scale ; (ii) to conduct pilot - plant studies on the distillation of low - temperature tars and collect commercially useful fractions.

Previous work : Erection of 3 tons/day pilot plant was completed. An oil - fired diesel oil distillation plant belonging to Andhra Pradesh State Road Transport Corporation was used for test distillation of 5,500 gallons of light tar. (Annual Report 1957-58, p. 66).

Experimental work and conclusions : (i) Heavy tar from low - temperature carbonisation of coal was distilled in a glass vacuum distillation unit up to a maximum vapour temperature of 300°C. at 10, 20, 30, 40, 50, 100, 200, 400 and 715 mm. pressure. The yieds of distillates up to 300°C. increased with decreasing pressure. At atmospheric pressure, the yield was 8.6 per cent and at 10 mm. it was 81.8 per cent. The softening point of the pitch residue also increased with decreased pressure. The softening point was 31.5°C. at atmospheric pressure and 81.5°C. at 10 mm. The percentage insolubles in pitch residues increased with decreasing pressure. However, when the insolubles were calculated on original dry tar basis, the values remained almost constant indicating negligible cracking of tar.

(ii) The 3 tons/day capacity tar distillation pilot plant was installed and pipe lines for water, air, gas, steam, tar and oil provided. Component parts such as air blower, burner assembly, instrument control panel, oil storage tanks and lifting post for the distillation column, etc., were fabricated. The plant was put into operation in the beginning of 1959. Four batches of light tar were distilled. The performance of each unit of the plant and properties of the distillates are being studied.

Future work: (i) Pilot plant studies will be conducted on the distillation and fractionation of heavy and light tar and on the preparation of road tar, pitch and creosotes. Vaccum distillation will also be studied. (ii) Laboratory studies on the distillation characteristics of low - temperature tar will be continued.

26. 8. Chemical examination of tar acids from low-temperature tar oils and ammoniacal liquors

Object: To investigate the chemical composition of tar acids present in the tar oils and liquors obtained from low-temperature carbonisation of coal ; and to devise methods for separation of pure components.

Previous work: Crude tar acids, solvent - extracted from aqueous liquors, were distilled into fractions boiling between 76°-170° C. at pressures varying from 10 - 27 mm. The lower fractions were examined by gas - liquid and paper chromatography.

Using the azo dyes (sulphonic) of the tar acids and methyl ethyl ketone saturated with water, and *n*-butyl alcohol as solvent systems, the following compounds were identified by paper chromatography: phenol, *o*-, *m*- and *p*-cresols, 3 : 5, 2 : 5, and 2 : 4 xylenols and guaiacol. The *m*-cresol content in *m*-/*p*-cresol fractions was estimated (Annual Report, 1957 - 58 p. 65).

Experimental work and conclusions: In the sample of crude tar acids earlier used for distillation a high percentage (38 per cent) of pitch was noticed. This was considered undesirable and in the present work a fresh sample was taken up for fractionation and for identification and estimation studies.

Fractionation of tar acids: The concentrated tar acid extract from the 'Luwesta' extraction experiments was desolvented to

obtain crude tar acids. The phenols carried over with the solvent were recovered and mixed with the crude tar acids. The crude tar acids were depitched by rapid distillation up to 160° C. at 7-8 mm. pressure. The pitch yield was 7.3 per cent.

About 600 g. of the distilled tar acids were fractionated in a 'Towers' type packed column at 5 - 12 mm. and at a constant reflux ratio of 1 : 5. Thirty-four fractions were collected up to 122° C. at 4.5 mm. (82 per cent). The residue was rapidly distilled at 1-1.5 mm. giving a distillate, boiling 96° - 130° C. (9 per cent) and a residue of about 2 per cent on the charge. The loss and hold - up were about 8 per cent.

Paper chromatographic identification of fractions :

(i) Monohydric phenols:

The phenols were converted into their azo dyes, which were then chromatographed on Whatman No. 1 filter paper (impregnated with 4 per cent Na_2Co_3 solution), using water - saturated methyl ethyl ketone and *n* - butanol (Annual Report 1957 - 58 p. 65); the fractions (boiling up to 85° C. at 6.5 mm.) were examined. A simple and a new technique for preparing the dye *in situ* on the filter paper was developed. Sulphanilic acid in 0.1 N concentration and excess phenol gave compact and clear spots.

Fractions 1 to 10 (b. p. up to 68° C. at 5 mm. and 47.6 per cent yield) contained mainly phenol with traces of *o*-cresol. Fractions 11 to 20 (b. p. 84° - 85° C. at 6.5 mm. and yield 19.1 per cent) contained mainly *o*- and / or *m*- cresol with traces of phenol, *p*-cresol, 2 : 6 xylenol and *o*-ethyl phenols. Fractions 21 to 26 (I batch, *ibid.*, p. 60) contained mainly 3 : 5 xylenol with traces of 2 : 4 xylenol.

This method was not satisfaceory for separation of the following pairs of substances : (1) *o*- and *m*- cresols (2) 2 : 5 xylenol and 2 : 3 xylenol (3) 3 : 5 - xylenol and *m*- ethyl phenol.

(ii) Dihydric phenols :

Major quantities of dihydric phenols were found to be present in the fractions above 140° C. at 27 mm. (I batch fractionation, *ibid.*) and 102° C. at 5 mm. Most of these were identified by using Whatman No. 1 filter paper (ascending technique) and solvent system benzene : AcOH : Water (3 : 3 : 31.5, v/v) mixture. A 5 per cent solution of the fraction in benzene was used for spotting. The spots

after development were visualised by using Folin Denis reage nt as spraying medium and subsequent exposure to ammonia vapours. Catechol, 3 and 4-methyl catechols, resorcinol, and 2, 4 and 5 methyl resorcinols were identified.

From the data of fractionation and paper chromatography it was noticed that the concentration of dihydrics was about 10 to 11 per cent.

Further work is in progress.

Column chromatographic studies : In order to estimate the individual dihydrics, work on partition column chromatography with U. V. spectrophotometry was taken up and is in progress.

Future programme : (i) To continue work on estimation of tar acids from liquors by column and gas-liquid partition chromatography; (ii) to take up work on tar acids from tar for fractionation, identification and estimation.

26. 9. Chemical examination of neutral oil from light tar

Object : To investigate the chemical composition of the neutral oil fractions.

Experimental work : Neutral oil prepared from the light tar by repeated alkali and acid washing was subjected to vacuum distillation and separated into six fractions, b. p. 54°–140° C/5 mm. The colour of the fractions ranged from pale yellow to reddish brown. The first fraction boiling at 54°–62° C/5 mm. exhibited a paraffinic odour although the odour was not characteristic of the fractions until they were fractionated further. Each of these fractions is being further fractionated and these will be examined by chromatographic and spectroscopic methods.

Future work : As outlined above.

26. 10. Electrophoresis of low-temperature tar and its fractions

Object : To investigate the possibilities of separation of different components of low-temperature tar and its fractions in their

original condition without degradation, using electrophoresis and electrodialysis techniques.

Previous work: Very little published literature is available on the application of electrophoresis and electrodialysis for the investigation of coal tars. H.A. Sack (*J. recherches centre Natl. recherche Sci. Lab.* 1951, No. 16, 21; *Compt. Rend.*, 1947, *224*, 833-5) employed electrophoresis in the study of solutions of coal tar pitch. This technique does not appear to have been used so far for the separation of components of tar.

Experimental work and conclusions: Light tar (170°-360°C.) from low-temperature carbonisation of coal was subjected to electrodialysis using distilled water and other liquids such as alcohol, dilute alkali, etc., in the electrode compartments. The constituents migrating into the electrode compartments were solvent-extracted and identified by paper chromatography. As expected, a major part of the tar acids migrated towards the anode and tar bases to the cathode. The tar acids and bases were almost free from neutral oils. The lower boiling phenols were completely removed from the tar oil. Phenol, *o*- and *m*-cresols, 2-6 xylenol, 3- and 4-methyl catechols were identified among the tar acids separated.

Future work: Work will be continued on the various aspects of the application of electrophoresis and electrodialysis techniques to the study of chemical composition of low-temperature tar and its fractions.

27. BRIQUETTING

Briquetting of non-caking coals

Object: Preparation of briquettes suitable as direct fuel from the high ash non-caking coals of Singareni Coalfields, and their carbonisation to produce smokeless coke briquettes.

Previous work: Briquettes made from high-ash, non-caking coals and lime-tar binder, though mechanically strong, crumbled to powder on open grate combustion or when carbonised directly. Preliminary tests on pretreatment of briquettes showed that heating in open

steam at 80°C. for periods up to 20 hr. almost doubled the briquette strength, which was normally obtained only after fifteen days' storage at atmospheric temperature. Preliminary maturing tests showed that heating of the briquettes at 200°C. for 2 to 4 hr. in a current of air produced stronger carbonised briquettes (Annual Report, 1957-58, pp.66-68).

Experimental work and conclusions:

(a) *Preparation of briquettes*: Experiments to rapidly raise the raw briquette strength were continued using the methods mentioned earlier, i.e., (i) curing in air at 80°C., (ii) in air and steam in a humidifier at 68 per cent R.H. at about 80°C., and (iii) in water vapour at 80°C. without air. The results from (ii) and (iii) showed practically the same trend with a rise in strength of 60-70 per cent on the raw briquettes in 20 hr.

(b) *Curing, maturing and carbonisation*: Repeated tests on curing, maturing and carbonisation indicated that (i) although steam-curing alone increases the raw briquette strength, it did not produce a strong carbonised briquette; however, steam-curing followed by maturing in air at about 200°C., produced a carbonised briquette whose strength was 60 per cent more than that of the raw briquette; (ii) longer duration of steam-curing followed by maturing, however, gave a carbonised briquette practically as strong as the raw briquette.

The influence of air rate during the maturing operation on the strength of the carbonised briquette was also investigated. The briquettes were treated in a single layer over a perforated plate in an electrically heated retort. Air was passed through a distributor tube from below the plate. In one series (columns 1, 2 and 3 of the table below) the briquettes, after maturing were cooled and re-heated for carbonisation; in the other (columns 4 and 5) the intermediate cooling was avoided. Typical results of varying the air rate during maturing and the conditions of subsequent carbonisation showed that, generally, higher air rates during maturing give a weaker briquette coke, although the strengths of matured briquettes were not much influenced by the variation in air rates used. It was further found that the strength of the briquette coke was higher if heating and carbonisation followed maturing without the intermediate cooling.

INFLUENCE OF MATURING AIR RATES ON STRENGTH OF CARBONISED BRIQUETTE

	Briquettes cooled between maturing and carbonisation			*No Cooling*	
	(1)	(2)	(3)	(4)	(5)
Maturing tests					
1) Wt. of briquettes, g.	190	382	395	403	400
2) Point compression (p. c.) strength of raw briquettes, lb.	205	200	170	170	170
3) Rate of heating, °C/min.					
up to 100°C,	2.0	1.0	1.0	2.5	3.0
up to 200°C.	1.5	2.5	1.5	1.47	1.47
4) Rate of air, cft/lb.hr.	13.6	10.22	9.18	9.26	6.03
5) Yield of matured briquettes, %	93	91.4	93	—	—
6) P. C. strength of matured briquettes	120	116	110	—	—
Carbonisation tests					
7) Rate of heating, °C/min.					
up to 100°C.	3.0	3.0	3.0	—	—
100° - 200°C.	2.4	2.4	2.4	—	—
200° - 600°C.	1.5	1.5	1.5	2.1	2.1
8) Yield of carbonised briquettes, %	82	85	77	77	76
9) P. C. strength of carbonised briquettes	205	220	195	240	325

Future work : Work will be continued to study the influence of all variables connected with pretreatment of briquettes ; pilot scale trials will also be conducted.

28. UTILISATION OF HIGH-ASH COALS BY GASIFICATION

Object : About two million tons of low rank high volatile non-caking coal is being mined annually in Andhra Pradesh. The probable reserves are estimated at 1350 million tons. Twenty per cent of the present production is slack coal (less than 1 in.) and the production of shaly coal is 3 per cent (ash 35 per cent). Utilisation of locally available high-ash coals, particularly slacks produced during mining, for the production of synthesis gas and fuel gas with a view to conserve high grade coals is of great importance.

Work during the year : A 25 Nm^3/hr. capacity oxygen plant is under installation for experimental studies on gasification of coals.

Future work : (i) Construction of an experimental coal gasification unit for the operation under pressure and slagging conditions, if necessary; (ii) study of fusion characteristics of ash from locally available coals; and (iii) study of the influence of operational variables on the course of gasification and quality of gasification products.

Heavy Chemicals and Fertilizers

29. ACTIVE CARBONS

29. 1. Production and development of active carbons

Object : To prepare active carbons suitable for bleaching of (i) vegetable oils, (ii) glycerine, chemicals and pharmaceuticals, and (iii) for solvent recovery and vapour adsorption.

Introduction: Active carbons prepared from high ash mineral coal from Singareni were effective in bleaching vegetable oils, pharmaceuticals, etc. Experiments were conducted on laboratory and pilot plant scale to produce grades of active carbons suitable for bleaching of different kinds of oils and other chemicals. The active carbons produced in this laboratory are registered under the trade name "HYKOL". Three grades, *viz.*, (i) HYKOL 'O' (Active carbon grade I),

(ii) HYKOL 'X' (Active carbon Grade II), and (iii) HYKOL 'BC' have so far been produced for bleaching purposes

(1) HYKOL 'O' (Active Carbon Grade I)

Previous work: The production was carried out for commercial purposes. Forty five tons of Hykol 'O' packed in 40 kg. polyethylene-lined jute bags were supplied for sale to Messrs. I.A.E.C., Bombay, the sole distributors. With a view to meet the increased requirements a grinding plant of capacity 0.5 tons/hr. was procured.

Medicinal quality castor oil passing BSS colour requirements was obtained using Hykol 'O' in conjunction with activated earth.

Present work : The installation of the grinding plant was completed. The plant was run at different settings of the classifier to determine the particle size of the product at each setting. A bucket elevator for feeding the raw material has been designed.

The production of Hykol 'O' during the year was 25.562 metric tons and the sales were 29.720 metric tons. In addition, 204 kg. were supplied to various industries and research departments for trial purposes.

The commercial exploitation of the patent of Hykol 'O' has been taken over by the National Research Development Corporation of India, New Delhi. Messrs. Voltas Ltd., Bombay, have been appointed as the sole selling agents from October 1958.

The filtrability and oil retention of Hykol 'O' were found comparable to the commercial active carbons.

(2) HYKOL 'X' (Activated Carbon Grade II)

Previous work: Hykol 'X', suitable for bleaching glycerine, sugar solutions, pharmaceuticals, etc., was prepared. Based on the preliminary experiments, the pilot plant was modified from gas to oil firing to an activation temperature of 850° to 900°C. In order to attain good heat transfer inside the reaction zone, it was decided to adopt direct firing with a fire-brick reaction zone. Experiments were planned to make use of direct firing in a refractory-lined rotary kiln.

Present work : Steam activation was carried out in refractory-lined oil-fired batch type rotary kiln varying the temperature, time of activation and feed size. A suitable product was obtained from semicoke of size $-\frac{1}{4}$" + $\frac{1}{8}$" activated at 900°C. for 2½ hours. Samples of the product tested were found suitable for bleaching castor oil, glycerine and glucose. Since January 1959 the steam activation plant is continuously being run three shifts per day to produce about one ton of Hykol 'X' per month. About 5 tons of Hykol 'X' were produced during the year of which about 400 kg. were supplied to various industries for factory trials.

Hykol 'X' thus produced was found to lose its bleaching activity during the grinding operations using equipment like Bantam Micropulveriser, pot mill, ball mill (wet and dry), edge runners, and stone mill (manual). The molasses and methylene blue decolorisation values of the sample ground in the above equipment were always less than those given by the sample hand-ground in pestle and mortar. In the above grinding equipment the ground material is further subjected to impact, which can be avoided in close circuit grinding system where the ground material is continuously classified and separated from the unground material. Experiment on these lines are underway.

An internally heated fluidised bed pilot reactor is being installed for activating the ground semicoke avoiding the necessity of grinding the coke after activation. It is expected that the fluidisation technique will result in better performance and more uniform product.

To evaluate its keeping quality, Hykol 'X' was stored at 60 per cent humidity under various temperatures, and at 40°C. under different humidities. The sample stored at 40°C. and 60 per cent humidity and 8.6 per cent moisture gave the maximum molasses decolorisation value calculated on dry basis.

(3) HYKOL 'BC' :

Hykol 'BC', a new grade of active carbon suitable for decolorisation of dark coloured castor and groundnut oils, when used in conjunction with activated earth was developed. About two tons of Hykol 'BC' were prepared. Samples (73 kg.) were supplied to various industries for trials. Results of the trials are awaited.

The average analysis of Hykol 'BC' was as follows: moisture 2-5 per cent; ash content 35-40 per cent; pH - 7; bulk density 0.65-0.70; and particle size, +170 mesh B.S.S., 10-15 per cent, - 170 +300, 35–40 per cent, and - 300, 45-55 per cent. Results of the decolorisation tests with castor oil on Y+5 R basis of Lovibond 1" cell) are shown below:

Original colour of castor oil 9.0 Y & 1.3 R

Type and quantity used (by wt. on that of oil)		Colour removal
Earth	Carbon	
Tonsil Optimum 1%	Hykol 'BC' O.25%	77%
Tonsil Optimum 1%	Darco S 51 O.25%	77%

(4) Active carbons for vapour adsorption

Previous work: Active carbon prepared by steam activation at 900° – 920°C. for varying periods, from L.T.C. coke obtained from Palana (Rajasthan) lignite were tried for adsorption of benzene and acetone. The sample obtained on activation for 1.5 hr. gave the maximum adsorption value.

Experimental work: Singareni coal fines were washed with sulphuric acid (1. 3 sp. gr.). The floats having less ash content were carbonised at 600°C. and the coke thus obtained was steam activated at 900°C. The product tested for adsorption of benzene gave a value of 54 per cent on the weight of carbon. Semi-coke obtained from Singareni coal after washing and steam activation yielded a product which gave a very low benzene adsorption value of 12 per cent on the weight of carbon.

Future work: (i) Production of Hykol brands of active carbons will be continued. (ii) Production of active carbon by steam activation in a fluidised bed reactor, and (iii) preparation of active carbon from coconut shells.

29. 2. Physico-chemical studies

Object : To determine the physico-chemical properties of active carbons with a view to correlate these with their activity and to establish the characteristics of good quality carbons for different purposes.

Previous work : The X-ray studies of coal, coke and active carbon were undertaken which have shown that the volume of mineral matter remained unaltered during carbonisation and steam activation and that the carbon in the active carbon was present in the form of small graphite crystals and the mineral matter consisted mainly of sillimanite and quartz. In the active carbon sample used in this study the weight ratio of mineral matter to carbon was 1:1; the volume ratio also worked out to be 1:1. The data were used for calculating the true volume of active carbon.

Experimental work and conclusions :

X-ray diffraction studies : X-ray diffraction patterns for two coals, two cokes and four active carbons were taken using the powder method of X-ray analysis. Both Kothagudem and Jambad coals gave weak patterns consisting of quartz and kaolin lines in which quartz lines were more prominent. The cokes gave patterns of greater intensity due mostly to quartz, some aluminium silicate and aluminium oxide.

Active carbons prepared in the Laboratary by steam activation of Kothagudem coke gave a pattern due almost completely to quartz and traces of sillimanite. Darco S $^{-51}$ gave a weaker pattern showing the presence of kaolin and some quartz. The pattern for Narbada carbon showed small amounts of mineral matter consisting of calcite, alumina and quartz whereas the pattern for carboraffin showed no lines, and hence absence of mineral matter. Steps are being taken to measure the intensity of the lines by microphotometer.

X-ray diffraction patterns of two grades of semicoke, A and B (A = above 1" size, and B = 1/2" to 1" size), obtained by low-temperature carbonisation of Jambad coals at 650°C. and 750°C. were taken. The pattern of grade A obtained at both the temperatures showed the presence of hydroxy apatite, quartz and kaolin.

The pattern of grade B obtained at 650° C. did not have the lines due to kaolin nor the strong lines of quartz, whereas the pattern obtained at 750° C. contained no lines of hydroxy apatite and kaolin but showed strong lines due to quartz.

Volumenometer : An all - glass apparatus to measure the true densities of porous substances was designed and fabricated. The apparatus can be used from 50 mm. to 760 mm. of mercury pressure. The true densities of standard non-porous substances like sodium chloride, potassium chloride and potassium dichromate were determined using nitrogen. The values when compared with the International Critical Tables showed a difference of 0.25 per cent. Nitrogen and hydrogen were found unsuited for use with *porous substances*. Helium gas which is not adsorbed by porous substances is being procured.

Surface area and porosity : Surface area of Hykol 'X' was calculated using data on adsorption of methylene blue and benzene. Porosity was calculated (a) from the methylene blue and benzene adsorption data, (b) from the apparent density obtained by mercury displacement method, and true density calculated from actual volume occupied by carbon and mineral matter as given by X - ray data.

The values of surface area of Hykol 'X'obtained from adsorption of benzene as well as methylene blue were nearly equal. The surface area was also found to be proportional to the stage of activation as determined by loss in weight.

Apparent density (by mercury displacement) of Hykol 'X' in granular form (- 1/4" to + 1/8") was determined to be 1.296 g. per ml. and true volume calculated by X - ray method was 0.41 ml/g. From these values the pore volume obtained was 0.36 - 0.37 ml/g. of carbon.

The values of porosity obtained from methylene blue and benzene adsorption were 0.37 and 0.36 ml/g. which are in agreement with that obtained by the X - ray data.

Pore diameter ; The average pore diameter calculated from benzene and methylene blue adsorption value was about 20 A°.

Future work : Work on the determination of active surface area porosity and pore size distribution and X - ray studies of active carbons will be continued.

30. BLEACHING EARTHS

30. 1. Rajasthan earths

Object: (i) To carry out pilot plant trials on activation of Rajasthan earths: (ii) to study the effect of various factors on the quality of the finished product.

Previous Work: Pilot plant experiments were continued on Mudh earth. In all sixteen batches (six on pilot plant and ten on bench scale) were worked which have shown that the particle size, quantity of the sulphuric acid used, concentration of the acidulated slurry, extent of washing, quality of water used for washing the acid-treated clay, and heat-activation had, individually, a considerable influence on the finished product. The Indoka-bala earth sample was analysed, activated and tested (Annual Report 1957-58, pp. 74-75).

Experimental work and conclusions: Pilot plant experiments on the activation of Mudh and Indoka-bala earths were continued. Dry grinding of the raw material for activation did not show any marked difference with the wet grinding, hence the latter was adhered to for preparing the material for acid activation.

Well water with 450 p. p. m., and municipal water with 250 p. p. m. total solids, and distilled water were used for washing the acid-treated earth to make it free of acid. The samples washed with municipal water to pH 3.1-3.3 gave a product with fairly good colour adsorption capacity. It was, however, observed that the decolorising capacity of the activated earth for groundnut oil was nearly 3 per cent more when distilled water was used instead of municipal water for washing.

Activated earths obtained in the above experiments did not possess satisfactory filtrability for alkali refined groundnut oil. Attempts made to improve the filtrability by incorporating 3-10 per cent of filter aids like washed sand of varying mesh sizes and a diatomaceous earth from Rajasthan did not give

better results. Improvement in filtrability was, however, observed by reducing the content of fines in the final product by controlled grinding in a ball mill, but there was a slight reduction (nearly 3 per cent) in colour adsorption efficiency. On the basis of the above study it has been concluded that using a good quality water and under appropriate conditions of grinding, an activated earth comparable to the Fulmont type can be obtained from the Mudh earth.

A report covering necessary details of the requirements and the conditions of processing 5 tons/day of Mudh fullers' earth was prepared and sent to the Rajasthan Government.

Future work: Fundamental studies on the above earths will be taken up.

30. 2. Mysore earths

Object: To carry out pilot plant trials on activation of earths from the deposits in Chincholi Taluk, Gulbarga Dist. (Mysore State).

Introduction and past work: Preliminary investigations on earths from various deposits in Chincholi Taluk had given encouraging results. Prospecting report from the Department of Geological Survey, Government of India, indicated that the quantities available there would be sufficient for commercial exploitation. Hence, a scheme for pilot plant activation of these earths in this laboratory was prepared and was sanctioned by the Government of Mysore. Sufficient quantity of fresh Korvi earth collected from four different pits was brought to the Laboratory for trials (Annual Report 1957-58, p. 76).

Experimental work and conclusions: Samples of the Korvi earth from four different pits and one mixed sample prepared from the pit samples were used in the present investigation. The above five samples were activated (i) by simply heating at 340°C., (ii) by treating with hydrochloric acid (5N) and heating the sample after washing at 340°C., and (iii) by treating with sulphuric acid (33 and 50 per cent w/w) and heating the samples after washing at 340°C. The activated earths thus prepared were tested for colour adsorption capacity and filtrability by standard methods.

It was observed that the natural Korvi earth had good colour adsorption capacity for groundnut oil, which was enhanced by heat

and acid activation. Typical colour adsorption using 1 per cent earth and its filtrability data are given below.

Description of the earth used	*Colour adsorption (%)*	*Filtrability (minutes)*
Natural Korvi earth	39.0	13
Korvi earth heated 340°C.	50.6	—
Korvi earth treated with HCl and heated at 340°C.	63.0	12
Korvi earth treated with H_2SO_4 (50 per cent) and heated at 340°C.	78.4	20
Tonsil Optimum	78.4	12

The time of contact to obtain maximum bleach was only five to ten minutes.

Future work: (i) Pilot plant experiments will be continued. (ii) Fundamental studies on the clays will be undertaken.

30. 3. Porbandar clays

Introduction: The samples of Porbandar clays were sent to this laboratory by Messrs. Sagar & Co., Porbandar, at the suggestion of the National Chemical Laboratory, Poona. It has been stated that huge deposits of fullers' earth are found near Porbandar area over a few hundred acres. The bed thickness and overburden, varies from 3 to 80 ft. and 2.5 to 4 ft., respectively,

Experimental work and conclusinns: Four samples from different localities near Porbandar were investigated by processing them with (i) hydrochloric acid (5 N) and (ii) sulphuric acid (33 and 50 per cent w/w).

The test results for one of them are as below :—

Acid used for activation	*Colour reduction (%)*	*Filtrability (minutes)*
1. Hydrochloric acid (5 N)	61.7	16
* 2. – do –	62.9	—
3. Sulphuric acid (33%)	50.6	17
* 4. – do –	69.1	—
5. Sulphuric acid (50%)	70.3	—
* 6. – do –	75.0	—

Note:— Serial numbers marked with an asterisk were heated at 340°C. after acid activation.

Future work : Work on these investigations has been completed.

30. 4. Activated earths for bleaching of mineral oils

Object : To develop suitable activated earths for bleaching of mineral oils and for reclamation of used mineral oils.

Previous work : Preliminary work has shown that bentonitic clay from Srirangapur, about thirty miles from Hyderabad, can be used after sulphuric acid activation for bleaching of lubricating oil (Annual Report 1957-58, p.76).

Experimental work and conclusions :

1. *Bleaching of lubricating oils* : Samples of earths obtained from Korvi (Mysore) and Srirangapur (Andhra Pradesh) were prepared in the Laboratory for trials on bleaching of lubricating oil. The following samples of earth, raw as well as processed, were tried for evaluating their bleaching capacity : (i) raw earth, (ii) earth activated with hydrochlroic acid (5N) or sulphuric acid (50 per cent), (iii) acid-activated earth kept overnight with aluminium sulphate solution (of four different strengths), then washed with liquor ammonia and

heated at 200°C., (iv) raw earth kept overnight with aluminium solution, then washed with liquor ammonia and heated at 200°C., and (v) Raw earth heated at 200°C.

Trials on bleaching of the raw lubricating oil containing 40 yellow and 2.1 red units, supplied by Messrs. Assam Oil Company Limited, were done using 7.5 per cent earth on the weight of oil at 85°C., for thirty minutes. The bleached oil was then kept at 140°C., for three hours for colour reversion test and the colour was determined using a $\frac{1}{4}$" cell. Typical results obtained are tabulated below.

	Sample	Residual colour of oil		Colour of oil on reversion	
		Y	*R*	*Y*	*R*
1.	Raw Srirangapur earth	30	1.4	40	9.2
2.	Raw Korvi earth	30	0.6	40	3.4
3.	Srirangapur earth activated with H_2SO_4(50%)	30	0.7	40	3.1
4.	Korvi earth activated with H_2SO_4 (50%)	30	0.7	40	3.1
5.	Activated Srirangapur earth treated with $Al_2(SO_4)^3$ solution (10%), washed with liquor ammonia and heated at 200°C.	30	0.7	40	2.6
6.	Activated Korvi earth treated as in (5) above	30	0.6	40	2.4
7.	Imported earth	30	0.8	40	3,6

The activated earth treated with aluminium sulphate showed minimum colour reversion. The colour reversion was less when the acid activated earth contained equilibrium moisture, about 14 per cent.

Samples were supplied to the Assam Oil Comptny Limited for testing. It is reported that the acid activated Srirangapur earth was similar to the currently used imported fuller's eaath, but, that the bleached oil was less stable to heat and storage. However, they have felt that at higher earth dosage it would give greater stability to the oil. The washed earths were found satisfactory for bleaching acid-treated wax; imported earths are being used at present for this purpose.

B. Reclamation and purification of used lubricating oils

Activated earths are used for reclamation and purification of used lubricating oils which contain dissolved water and oxidation products. It is necessary that the clay for this purpose should have water repelling property. It has been reported that activated earths now manufactured in this country are not suitable as these form a slurry with the water present in the oil. The estimated 'requirements of these earths in the country are about 250 tons per year.

While working on the naturally occurring bentonitic clays from Korvi (Mysore), it was observed that it has got the required properties which are further improved by heat activation. The clays can be used for removing the suspended impurities and improving the colour of the used lubricating oils. The following reading compare the results obtained with different samples of activated earth for purifing the used oil.

Sample	*Residual colour of the* ($\frac{1}{4}$" *cell*)	
	Y	R
Raw Korvi earth	40	26
Heat activated Korvi earth	30	24
Imported earth	40	26
Colour of the reclaimed oil supplied	40	24

Samples of the earths prepared in the Laboratory have been supplied to a party dealing in reclamation of used lubricating oils for tests and report.

Future work: Work on improving the characteristics of raw Korvi earth will be continued so as to make it superior to the currently used fuller's earths.

Note: Investigations on this problem were undertaken at the instance of the Ministry of Steel, Mines and Fuel, Government of India.

30. 5. Survey of fullers' earths and bentonitic clays in India

Object: (i) To obtain information about the occurrence and availability of fullers' earths and bentonitic clays in India; (ii) to obtain samples of earths and clays from various deposits, for preliminary investigations on their suitability for bleaching vegetable and mineral oils.

Work during the year: A questionnaire was addressed to the Directors of Mines and Geology departments of all the States in India and other sources with a request to furnish information on the occurrence, availability, approximate details of the deposits, and the procurement of these raw materials for last three to five years.

The information collected on (i) occurrence and availability and (ii) chemical analysis is summarised below. Several samples of fullers' earths and bentonites from various sources are being collected for investigations under a research scheme sponsored jointly by the Indian Central Oilseeds Committee and the Council of Scientific and Industrial Research.

(i) Occurrence and availability of fullers' earths and bentonites:

Location	*Description of the deposit*	*Estimated reserves (tons)*	*Remarks*
A. *Bombay.*			
(1) 40 miles from Murtuzapur in Vidarbha	deposit in 2-pockets at depths of 5-15 ft.; thickness of bed 3-5 ft.; and 2 ft. of earth mixed with quartz lumps.	10,000	
(2) $2\frac{1}{2}$ miles from Lakhanaka in Bhavnagar (Saurashtra).	Lithomarge clay partly white, soft and grit free; sometimes greenish associated with variegated or lavender coloured clays; the bed thickness of the earth is 10–20 feet.	extensive; detailed prospecting to be done.	commercially exploited.
(3) Near Nandana Ran and Mewasa in Nayanagar (Saurashtra).	fullers' earth in association with ochres and laterites.	—	—
B. *Madras.*			
Vallam, Kannatangal, Mappedu, Killacheri and Koppur in Chingleput and South Arcot districts.	clays are mixtures of montmorillonites and illite; depth over 15 ft.	5 million.	

Location	*Description of the deposit*	*Eastimated reserves (tons)*	*Remarks*
C. *Andhra Pradesh.*			
(1) Near Marepalli, Tandur Taluk.	grey soft earth, also reddish under 1-4 ft. depths: thickness of the beds 3-6 ft.	extensive	exploited for commercial purposes.
(2) Near Sriran-gapur in Pargi Taluk.	bentonite mixed with silica and other grit material.	1,80,000	do
D. *Bihar.*			
In several areas of Santal Pargana district.	—	—	A few deposits exploited,
E. *Kashmir.*			
(1) Budil area, Tehsil Rajouri.	an alate formation of the older rocks soft-soapy, white to bluish white; 6-7 ft. thickness of bed.	—	—
(2) Bhimber, Rattanpur, Thera, etc.	white pink and greyish white earths.	—	—

(ii) Chemical analysis of some of the earths reported is shown in the table below:

NAME OF EARTH	CONSTITUENTS ANALYSED (%)							
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MgO	CaO	alkalies	moisture
Vidarbha earth	50.8	6.27	10.39	1.2	2.69	1.67	—	9.77
Lakhanaka earth	58.4	15.81	3.1	—	4.12	1.19	2.37	14.95
Budil earth	58.58	17.2	8.2	—	0.29	7.1	—	7.2
Bhimber earth	48.64	—	—	—	8.67	1.8	—	29.91

Future work : To obtain further samples and information and conduct preliminary studies on the lines stated above.

31. INORGANIC CHEMICALS

Introduction : To develop suitable industrial methods for the preparation of some inorganic chemicals of interest to the industry.

The work under this project is at present limited to preparation of hydrazine sulphate, dessicants, and pigments.

31. 1. Preparation of hydrazine sulphate

Previous work : Hydrazine hydrate prepared on laboratory scale by oxidation of ammonia with sodium hypochlorite was reacted with sulphuric acid to give hydrazine sulphate. The synthesis of hydrazine hydrate was done at 85 °C. and atmospheric pressure instead of 160 °C. and 30 atmospheres in the modified Raschig process.

Experimental work and conclusions : Pilot scale experiments were done using the all-glass chlorination plant on the preparation of hydrazine in three stages; (a) preparation of sodium hypochlorite at 5 °C., (b) oxidation of ammonia to hydrazine hydrate by sodium hypochlorite, in the presence of glue, at 85 °C. and atmoshperic pressure, and (c) conversion of hydrazine hydrate into hydrazine sulphate at 5 °C. The yield of hydrazine sulphate was about 90 per cent on the basis of chlorine used. Excess ammonia was recovered as 20 per cent liquor ammonia.

Estimated cost of production calculated for 10 kg. of hydrazine sulphate per batch works out to Rs. 6.71 per kg. The current market price of the chemical is Rs. 16 per kg.

Future work: The work outlined above will be continued. Preparation of hydrazine hydrate will be undertaken.

31. 2. Dessicant from gypsum

Introduction: Drierite, a dessicant prepared from gypsum is used for drying gases and solvents such as chlorine, ethers, and hydrocarbons. It can be easily regenerated for reuse.

Experimental work: A satisfactory sample of dessicant was prepared by heating gypsum at 220° - 250°C. The product was tested by measuring the rate of absorption of moisture at different relative humidities and time.

31. 3. Preparation of barium chemicals from barytes

Object: To develop suitable methods for the manufacture of barium pigments and other barium chemicals from barytes.

Previous work: Experiments were done on reduction of barium sulphate to barium sulphide using lignite as reducing agent (Annual Report 1957-58 pp.78, 79).

Experimental work and conclusions:

(a) Barytes with a barium sulphate content of 99.4 per cent, and semi-coke and pitch obtained by low-temperature carbonisation of Kothagudem coal, were used. The conversion of barytes to barium sulphide by heating barytes with (i) semi-coke and (ii) pitch, in the proportion of 1 : 0.2 by weight at 950°C. for two hours was 54 per cent and 90 per cent respectively. Using barytes and semi-coke in the proportion of 1 : 0.3 the conversion was 84 per cent. The conversion to barium sulphide was completed by passing water gas over barytes at 950°C. for two hours.

(b) *Blanc fixe from barium sulphide*: Blanc fixe was precipitated from barium sulphide solution by sodium sulphate. The product was not free flowing as required by the industry. Further trials are in progress.

Barytes powder: A snow white sample of barytes from Andhra Pradesh was powdered in a colloid mill. Absorption of double distilled linseed oil and particle size of barytes powder determined according to the Indian Standards Specification I.S. 33- (1950), were comparable to a commercial grade blanc fixe sample.

Future work : (i) Work on blanc fixe will be continued. (ii) Work will be undertaken on lithophone, and barium chemicals.

31. 4. Cadmium sulphide (orange)

Object: To develop suitable method for the manufacture of cadmium sulphide (orange).

Experimental work and conclusions: A process for the preparation of pure cadmium sulphide (orange) from cadmium metal was evolved The product was tested and found satisfactory as a ceramic pigment. The cost of production of cadmium sulphide by this process was Rs. 14.50 per kg. (cf. market price of Rs. 18 per kg). A note on the requirements of equipment and estimated cost of production for 100 lb. per day cadmium sulphide (orange) has been prepared.

Future work: The work has been completed.

32. SULPHUR

32. 1. Utilisation of gypsum and other sulphates

Object: (1) To evolve an economic process for the production of sulphur from gypsum; and (ii) to study processes for utilisation of gypsum and other sulphates in the preparation of sulphuric acid and sulphides etc.

Previous work: Work was carried out on the production of hydrogen sulphide from gypsum, sodium sulphate and barium sulphate. Pilot plant experiments on the production of calcium sulphide from gypsum were carried out (Annual Report 1957-58, pp. 78-79).

Experimental work and conclusions:

Hydrogen sulphide from gypsum: Fifteen kilograms of gypsum-coke and gypsum-lignite mixtures in the form of pellets of size 1.2-1.5 cm.

were heated at 900°C. in a vertical cast iron reactor. Steam was passed over the mixture at 900°C. (a) from the commencement of the reaction, and (b) after heating for two hours to allow for the reduction of gypsum to calcium sulphide. The yield of hydrogen sulphide was, however, same in the two cases.

The conversion of gypsum to calcium sulphide was 90 per cent in two hours when the reactants were taken in the theoretical proportion, mixed thoroughly and heated at 900°. Some observations made on the reduction of gypsum by coke or lignite were (i) that the reduction was faster with lignite than with coke, (ii) the optimum proprotion was 1 : 0. 4 in case of lignite and 1 : 0. 2 in case of coke and (iii) calcium sulphide was oxidised to calcium sulphate if it came into contact with air at the high temperature.

The evolution of hydrogen sulphide, however, stopped after two hours, only at a 38 per cent conversion stage due to the pellets becoming hard and impervious to further action of steam. The cause of hardening of the pellets is being investigated.

Future work : Pilot plant investigations to establish the optimum conditions for the production of sulphur from gypsum and to ascertain the economic feasibility of the process.

31. 2. Utilisation of iron pyrites

Object : To evolve processes for utilisation of iron pyrites in the preparation of sulphur, sulphuric acid, sulphides. etc.

Previous work: Experiments were carried out on the preparation of (a) sulphur and (b) iron sulphide, using Ingaldhal (Mysore) iron pyrites with a sulphur content of 23 per cent. After separation of the magnetic portion, steam was passed over the non - magnetic fraction of the pyrites in the presence of coke. Eighty-three per cent of sulphur present was liberated as hydrogen sulphide, and 11 per cent as elemental sulphur. The conversion of iron pyrites to iron sulphide by heating pyrites and coke to 800° C. was 87 per cent of the pyrites (Annual Report 1957–58, p. 79).

Experimental work and conclusions:

Sulphur from pyrites: A pre-design cost estimate was made. It is found that sulphur obtained by this method would be more expensive than the imported brimstone.

A detailed study of various possibilities of utilising the indigenous pyrites has been made and it is concluded that manufacture of sulphuric acid from pyrites will be more economical than to prepare sulphur from it.

Sulphuric acid from pyrites : At a suggestion made by the Laboratory the Government of Mysore are considering a proposal to set up a factory for the manufacture of sulphuric acid using Ingaldhal iron pyrites. A cost estimate for the production of 25 tons per day sulphuric acid from Ingaldhal pyrites has been supplied to them.

Iron sulphide from pyrites : Iron sulphide with 35.2 per cent sulphur was prepared by wet grinding of Taradevi (Himachal Pradesh) pyrites to 300 mesh B.S.S., and heating at 930 – 950°C. in the absence of air. The commercial iron sulphide contains only 20 – 25 per cent sulphur.

Future work : No further work on iron pyrites is contemplated for the time being.

33. FERTILIZERS

33. 1. To keep contact with, study and offer proposals for establishment of fertilizer industry in India

Object : To make proposals for the manufacture of suitable fertilizers at appropriate locations in the country, using economical processes and available raw materials.

Previous work : A detailed note on the technical and economic aspects of installing a nitrogen fertilizer factory having a capacity of 70,000 tons of nitrogen per year near the coal mines at Kothagudem (Andhra Pradesh) was prepared and sent to various industrialists and the Governments of India and Andhra Pradesh.

A report was prepared for Messrs. Hyderabad Chemicals and Fertilizers Ltd., Hyderabad, on the manufacture of high analysis complex fertilizers, namely ammoniated triple superphosphate (Ammophos) and nitrophosphate (Nitrophos) containing nitrogen and P_2O_5 in the ratio of 20:20 (Annual Report 1957-58, p.82).

Work during the year:

A. Mixed fertilizers for Andhra Pradesh:

The modern trend is directed towards the production and consumption of high analysis and complex fertilizers. Such fertilizers provide the farmer with a balanced and ready-to-use product containing both nitrogen and phosphorus. The high concentration of nutrients in the product results in reducing the freight charges per unit of the fertilizer, making it more economical.

A report entitled "Suggestions for schedules of production and location of the proposed fertilizer factory in Andhra Pradesh," has been prepared to be submitted to the Technical Committee on Fertilizers. Government of India, during their visit to Andhra Pradesh in April 1959.

A note on the alternative schedule of production of mixed fertilizers at Kothagudem has been prepared, which forms an appendix to the original note on the economic and technical aspects of the nitrogen fertilizer factory at Kothagudem.

The note suggests the following alternative schedules of production:

Alternative	*Production per Day (Tons)*	*Total Investment (Lakhs of Rupees)*
1. urea (45% N)	445	1804
2. i) urea (45%N)	222	
ii) kalk ammonium nitrate (21%N)	477	1994
3. i) urea (45% N)	225	
ii) ammonium sulphate (21% N)	280	
iii) diammonium phosphate (DAP) (21% N, 53% P_2O_5)	189	2135
4. i) urea (45% N)	222	
ii) nitrophos (20% N, 20% P_2O_5)	500	2042

Of the four schedules the first two are for straight nitrogen fertilizers, while the last two are for the high analysis complex fertilizers.

The note also discusses the three locations, *viz*, Vijayawada, Ramagundam and Kothagudem, with respect to different aspects like raw materials, utilities, transportation and movements of raw materials and products. Based on the discussions, Kothagudem was considered to be a suitable location.

Appendix I. Raw materials and finished products.

Appendix II. Estimated investment for the alternative schedules of production.

Appendix III. Ton-mile calculations giving the movement of raw materials and finished products in terms of ton-miles per day for Vijayawada, Ramagundam and Kothagudem.

Appendix IV. Tables containing the output of various qualities of coal in 1958-59 at Singareni Collieries Co. Ltd., along with the proximate analysis of coals produced by the company in January 1959.

Appendix V. Estimated cost of electric power generation at Kothagudem.

A note was also prepared for a private party on estimated capital investment and production schedule for a small fertilizer plant of capacity 24,500 tons of nitrogen per year, to produce concentrated complex fertilizers.

B. *Fertilizers from the by-products of caustic soda industry*:

There are numerous caustic soda plants mostly ranging from 5 to 25 tons per day capacity. The relatively small quantities of hydrogen obtained as by-products can be processed to produce ammonia. Also the hydrogen can be combined with chlorine to produce hydrochloric acid which can be used in the manufacture of dicalcium phosphate. This phosphorus fertilizer has been tried in our country and is reported to be as good as super-phosphate.

A scheme for the manufacture of dicalcium phosphate has been prepared. For this the source of hydrogen will be the by-product of a caustic soda plant having a capacity of 25 tons per day. The feature of the scheme is to use the hydrogen and chlorine to obtain about 23 tons of hydrochloric acid per day, and to treat imported rock phosphate with this acid resulting in the yield of about 53 tons per day of dicalcium phosphate dihydrate (41 per cent P_2O_5).

The scheme contains the following data regarding raw materials and products.

Raw materials (tons per day):

(1) Common salt (100 per cent NaCl) 37.0; (2) quick lime 100 per cent CaO/8.75; and (3) rock phosphate (32 per cent P_2O_5) 68. 7.

Products (tons per day) :

(1) Caustic soda 25.0; (2) chlorine 22.2; (3) hydrogen 0.625; (4) hydrochloric acid 22.8; and (5) dicalcium phosphate (41 per cent P_2O_5) 53.7.

Future work: (i) Necessary technical help will be given to the Government of Andhra Pradesh in procuring and installation of the proposed fertilizer plant, (ii) further technical work needed for the installation of small fertilizer plants to utilise the by-products hydrogen and chlorine from caustic soda industry will be carried out.

33. 2. Non-caking ammonium nitrate fertilizer

Object: The proposed fertilizer plant at Nangal is to manufacture ammonium nitrate. Being hygroscopic, ammonium nitrate tends to cake and creates difficulty in storage and handling. The object is to find suitable diluent and coating agent for ammonium nitrate to render it free flowing.

Previous work : Silt, bone and imported fatty amines were used as coating agents (a) for ammonium nitrate as such, and (b) for ammonium nitrate diluted with silt or limestone. The performance of silt and bone powder as coating agents was superior to that of the imported material.

Experimental work and conclusions: At the suggestion of the Nangal Fertilizers work was undertaken for determining the suitability of Sutlej river silt diluent and coating agent for ammonium nitrate. The silt consisted of 86 per cent of the material of size 30+100 mesh B. S. S., 8 per cent -100 +150 mesh, and the rest -170 mesh. Coarser fraction of the silt (-30× 100 mesh) was used as diluent. The finer fraction of the Sutlej river silt (-100×150 mesh) and also of Godavari river silt (sieve analysis: 20 per cent -100×150 and 80 per cent -150 mesh) were used as coating agents. Forty per cent of the silt on the weight of ammonium nitrate was used for dilution and 3 per cent for coating. The prepared samples were subjected to hygroscopicity test at 30°C. and 80 percent humidity. The results were as follows :

Samples	*Percentage of moisture absorbed in*			
	2 hr.	*4 hr.*	*6 hr.*	*24 hr.*
Ammonium nitrate diluted with Sutlej silt and coated with Godavari silt	0.97	1.47	1.87	12.60
Ammonium nitrate diluted and coated with Sutlej silt	0.98	1.49	1.90	13.36
Ammonium nitrate diluted with sutlej silt	1·04	1.70	2.10	19·80

The Sutlej river silt was found satisfactory both as diluent and coating agent. Nangal Fertilizers have agreed to conduct factory trials using the silt as coating agent.

A short report of the work done in the Laboratory on non-caking ammonium nitrate was prepared and sent to (1) Sindri Fertilizers and Chemicals Private Ltd., (2) Nangal Fertilizers and Chemicals Private Ltd., (3) National Chemical Laboratory, Poona and (4) National Research Development Corporation of India, New Delhi. It was suggested to use river silt instead of imported colloidal iron Kiesulghur and hydroxide, at present being used by the Sindri Fertilizers for coating the double salt ammonium sulphate nitrate fertilizer.

Future work: Further work will be planned on the basis of results of the factory trials and laboratory tests at Nangal and Sindri.

34. WHITE CEMENT

White cement from felspar

Object: To develop a process for making white cement from felspar.

Previous work: The National Research Development Corporation of India, Near Hutti have sanctioned a pilot-plant scheme on the project.

Bulk quantities of the raw materials limestone, gypsum and felspar for large scale experiments were obtained from different parts of the country. A pilot cement rotary kiln shell was obtained on loan from the National Chemical Laboratory, Poona (Annual Report 1957-58, p.84)

Experimental work and conclusions:

(a) *Preparation of white cement using Sithampundi anorthite felspar by single sintering method*:

Trials were conducted using Sankari limestone and Sithampundi felspar in a proportion of 3: 1. Hyderabad quartz was added as a partial replacement of Sithampundi felspar. The batches were made in the form of pellets which facilitated uniform heat treatment of the batch and quick cooling of the clinker. Heating schedule for clinkering was studied and the following gave the best clinker formation in the cements. The batches were heated slowly up to 900°C. in 2 – 3 hr., then heated at a rapid rate to reach the maximum temperature of 1450° – 60°C in 45 – 60 min. and cooled quickly to room temperature. The best cement in these trials was obtained using limestone, felspar and quartz in proportion of 76: 15: 9. This had a molecular composition 15 CaO. Al_2O_3.5 SiO_2. The setting time and crushing strength of this cement was comparable to good quality white cement, with the exception that its colour contained a greenish tinge. Sithampundi felspar contained 1.5 per cent of iron, manganese and chromium impurities, which could not be separated by washing, magnetic separation or froth

flotation. This felspar, therefore, can be used only for making light coloured cements.

(b) *Preparation of white cement using potash felspar from Rajasthan and Andhra Pradesh by double sintering method:*

Trials were conducted using felspar from Rajasthan and Andhra Pradesh; limestone from Rajasthan and Madras, and gypsum from Rajasthan. Ten kilogram batches were calcined at 900°C. for three hours and quenched in water. The water soluble alkalies were separated by leaching with hot water. The sludge thus obtained was calcined at 1430°C. in a gas-fired furnace, in high alumina crucibles prepared in the Laboratory, and cooled rapidly. The batches containing (i) felspar from Golconda, limestone and gypsum from Rajasthan, and (ii) felspar, limestone and gypsum from Rajasthan in 1: 3. 1 proportions gave good white cements whose properties are given below:

Type of cement	*Setting time*		*Crushing strength after two days of water curing (lb/sq. in.)*
	Initial	*Final*	
white cement (i)	30 min.	1 hr. 46 min.	1500
white cement (ii)	20 min.	1 hr. 40 min.	1250
white cement imported from Germany.	1 hr. 55 min.	over 3 hrs.	1600
white cement available in market.	38 min.	1 hr. 50 min	1350

The white cement samples prepared in the Laboratory were supplied to (1) Director of Mines and Geology, Rajasthan, (2) Industrial Liaison Officer, Jaipur, and (3) Regional Liaison Officer, Madras.

Pre-design cost estimate of the process has shown that the cost of production of white cement by double sintering process will be Rs. 170/- per ton. The prevailing prices of the indigenous and imported white cements in the market are Rs. 250 to Rs. 400 per ton.

Refractory lining of the pilot rotary cement kiln has been done and installation of the kiln completed, except the motor and reduction gear which are expected to be received from abroad shortly.

Future work: The installation of the rotary cement pilot plant kiln will be completed after receiving the required accessories. Experiments will be carried out on batches of 250 kg. per day varying the conditions such as feed rate, temperature, etc. The raw materials will be felspar from Andhra Pradesh and Rajasthan and limestone and gypsum from Rajasthan.

35. CATALYSTS

Catalysts for low-pressure hydrogenation of tar

Object: Preparation and evaluation of catalysts for low-pressure hydrogenation of tar obtained by low-temperature carbonisation (l.t.c.) of coal, for obtaining diesel fuel and lubricants.

Introduction:

Low-pressure hydrogenation of tar is usually carried out at 400°–450°C. and 100-200 atmosphere in the presence of a suitable catalyst. The processes for preparation of such catalysts are mostly covered by patents which do not give any details. The following catalysts supported on carriers have been used:

(i) metals like chromium, iron, tin, nickel and cobalt;

(ii) oxides of metals like aluminium, molybdenum, magnesium, chromium, iron and barium; and

(iii) sulphides of metals like iron, chromium and molybdenum.

Molybdenum sulphide or oxide supported on activated alumina or bentonite or fuller's earth as the carrier is recommended for this purpose, as these carriers have a pore radius of about 50-100 A° and will have a moderately high surface area and suitable pore size after the deposition of the catalyst inside the pores.

Experimental work: Based on the above considerations, preparation of molybdenum oxide catalysts supported on (a) activated

alumina and (b) fuller's earth from Korvi (Mysore) as carriers have been started.

Determination of surface area and pore volume of the carrier and catalyst and estimation of molybdenum in the catalyst by chemical analysis have been done. The prepared catalysts are being evaluated for their activity for hydrogenation.

Further work : (i) Work on evaluation of various molybdenum catalysts supported on (a) activated alumina and (b) activated earth will be continued; (ii) the catalyst found satisfactory will be used for hydrogenation of l.t.c. tar.

36. REMOVAL OF SILICA FROM SPENT LIQUOR FROM PAPER MILLS

Object: To remove the soluble silica from spent alkali solution so that a good quality calcium carbonate may be obtained during causticization of the alkali liquor.

Introduction: In paper mills the spent liquor (black liquor) after digestion of bamboo with sodium hydroxide, etc., is burnt in a furnace for the recovery of alkali salts. The alkali smelt consisting mainly of sodium hydroxide, sodium carbonate and some silica impurity, is dissolved in water (green liquor) and causticised with lime to convert the alkali carbonate to its hydroxide. During causticization, calcium silicate is precipitated along with calcium carbonate. Owing to its high silica content the sludge has to be discarded and every time a fresh quantity of lime stone has to be used to prepare quick lime for causticization, which is wasteful.

Although some work has been reported in literature on precipitation of silica from sodium silicate by the action of carbon dioxide, no systematic work on separation of silica from spent alkali liquor from paper mills appears to have been carried out.

Experimental work and conclusions: Alkaline solutions containing sodium hydroxide, sodium carbonate and sodium silicate were prepared having similar alkali and silica concentration as that of green liquor and carbon dioxide was passed into the solution at temperatures varying from 20° to 90°C. till the pH of the solution decreased to a value between 11.5 and 9.0 depending upon the temperature of the

experiment. About 90 per cent silica was precipitated which was washed free of adhering alkali salts. Eighty six per cent of silica was separated from green liquor at 60°C., with less than 1 per cent of the alkali in the precipitate. The results obtained are tabulated below :

Sl. No.	Composition of the alkaline solution (gm/litre).						Temp. (°C.)	Silica separated. %	Vol. of wash water x original vol.	Alkali with silica precipitate %	Nature of silica precipitated
	SiO_2	Na_2O	NaOH	Na_2CO_3	$NaHCO_3$	Na_2S					
I.	10.60	5.00	5.6	...	...	...	30	N. D.	...	7.4	gel form
II.	6.64	7.60	9.9	...	40.0	...	30	over 95.0	3	2.3	granular; settles down quickly.
III.	6.33	2.64	—	97.6	...	...	45	87.0	1.5	4.8	do-
IV.	6.33	2.64	—	100.0	...	...	60	84+6 *	3	0.8	do-
V.	6.33	2.64	—	100.0	...	...	90	65+18 *	3	1.0	do-
† VI.	5.90	66.13 **	—	—	...	5.46	60	86	-	—	do-

Future work : (i) Pilot scale experiments using 50 gallons of green liquor at room temperature and 60°C.; (ii) similar experiments using black liquor.

* Second precipitation at a lower temperature and at pH 9. 0.

** Total alkalies calculated as Na_2O.

† Green liquor of 120 g. of alkali smelt per litre concentration was used. The green liquor after silica precipitation was free from suspended solids. The silica precipitate occupied 20 per cent volume of the solution.

Ceramics

37. GLASS AND CERAMICS DEVELOPMENT

37. 1. Porcelain

Object: To develop body and glass compositions suitable for good quality porcelains.

Previous work: Suitable compositions were developed using raw materials available both within and outside Andhra Pradesh. The best specimens were fired at 1250°-1300°C. in an electric furnace. Since a reducing atmosphere could not be obtained at the finishing stages of firing in the electric furnace, the fired porcelain was not of the desired whiteness (Annual Report 1957-58 p.86).

Experimental work: The construction of the oil-fired ceramic kiln based on the design supplied by the Central Glass and Ceramic Research Institute, Calcutta, was completed, and a few trial experiments were conducted to test the performance of the kiln.

Future work: Suitable compositions will be developed for making saggers and the test pieces will be fired without coming in direct contact with the flame. The body and glaze compositions developed in the Laboratory will be then tried on a pilot plant scale.

37. 2. Brick clays from Bombay State.

Object: To study the various properties of the brick clays obtained from Bombay State with a view to assess their suitability in brick industry.

Experimental work and conclusions:

The work was taken up in February 1959. Twenty five samples of clays were received. They were crushed and ground in an edge runner mill. A representative sample of each clay weighing thirty pounds was obtained by the method of quartering and coning.

The following properties of the clays were studied: (a) water of plasticity, (b) linear dry shrinkage, and (c) linear fired shrinkage at 800°, 900°, 1000° and 1050°C.

The percentage of water of plasticity was determined using the Pfefferkorn plasticimeter. According to this method, the amount of water which is required to be mixed with the clay in order to reduce the height of a cylindrical specimen of the clay-water body by a third of its initial height under the impact of a constant load is calculated as the water of plasticity. On an average, at least five samples were used to determine the deformation at each water content.

The linear shrinkage of the samples dried at 110°C. and that of the samples fired at 800°, 900°, 1000° and 1050°C. was determined using " Kidney-shaped " specimens by the conventional method.

Future work: The following properties of the clays will be determined: (i) Absorption behaviour of the samples dried at 110°C. and of the samples fired at 800°, 900°, 1000° and 1050°C., and (ii) the ultimate crushing strength of the samples fired at the above mentioned temperatures.

The usefulness of the clays for the purpose of making bricks will be assessed after the above data are obtained.

37. 3. Titania opacified enamels

Object: To develop suitable enamel compositions possessing adequate gloss and resistance to acids, weathering, and mechanical and thermal shock. Titanium dioxide which is available in India is proposed to be used in commercially feasible enamel compositions in place of tin oxide which is an expensive and imported chemical.

Previous work: Nine enamel frits were prepared using the following compositions as the standard: SiO_2 49.8; Al_2O_3 3.6; B_2O_3 12.6; Na_2O 10.6; K_2O 0.6; Sb_2O_5 0.2; TiO_2 14.5; CaO 1.0; MgO 1.6; ZnO 1.2 and F_2 4.3 per cent. The SiO_2-content was varied at the expense of B_2O_3. The frits were melted at 1100°–1200°C., ground in pot mills with suitable mill additions and sprayed on ground-coated sheet steel samples, the cover coat thickness being 0.2 mm. after firing. The gloss of the enamels as well as the acid resistance as per the P. E. I. spot test were studied (Annual Report 1957–58, p. 88).

Experimental work and conclusions :

Four series of enamel cover coat frits containing titania in place of tin oxide were prepared. The raw materials used and the ranges of compositions are given below :

Raw materials	*Compositions (wt. per cent) in the various series.*			
	D	E	F	G
Felspar	0–4.0	4.0	4.0	4.0
Boarx	28.6	28.6	28.6	28.6
Quartz	37.5 - 42.6	29.7 - 40.7	38.7	33.9 - 37.1
Soda ash	0–1.9	—	—	2.8 - 8.5
Potash	0–0.7	—	—	—
Sodium nitrate	3.0	3.0	3.0	3.0
Cryolite	0–6.5	6.5	6.5	6.5
Sodium antimonate	0.2	0.2	0.2	0.2
Titanium dioxide	12.0	10.0 - 21.0	12.0	12.0
Aluminium hydroxide	0–2.2	1.0	1.0	1.0
Zinc oxide	1.0	1.0	0–2.3	1.0
Magnesium carbonate	3.2	3.2	0–5.6	3.2
Whiting	0–1.5	1.5	0–3.3	1.5
Sodium silico-fluoride	0–5.8	—	—	—

Four different enamels were prepared from each of the above series. The mill additions used were: frit 100 parts, sodium nitrate 0.23 parts, bentonite 0.23 parts, and Kundara china clay 3.00 parts.

Furture work : (i) The gloss and acid resistance of the above enamels will be studied after firing; (ii) after completion of the above work, investigations on the development of suitable compositions for glass-lined chemical equipment will be taken up.

37. 4. Standard pyrometric cones

Object: To develop compositions and a process for the manufacture of standard pyrometric cones for low and high temperatures.

Previous work: Staffordshire type cone 05a and Orton type cone 05 were developed and standardised.

Experimental work and conclusions: Work was continued on both Staffordshire and Orton type cones. Five different cones of the Staffordshire type and fifteen different cones of the Orton type, detailed below, were developed and standardised:

Staffordshire type : Cone Nos. 05 to 01

Orton type: Cone Nos. 010 to 02 and 5 to 10.

A frit containing china clay, quartz, whiting and borax was prepared and used for the development of sixteen out of the above twenty numbers of cones. The refractory cones (Nos. 7 to 10 of the Orton type) did not contain any frit and were made from mixtures of china clay, quartz, felspar and whiting.

The Staffordshire cones possessed a relatively poor green strength and were hence pre-calcined in saggers prior to testing.

An externally heated electric crucible furnace was used for testing the cones. The cones were heated to the end-point at the rate of 150°C. per hour.

The cones developed in the Laboratory compared very favourably with the imported Orton cones with regard to their thermal behaviour, the maximum variation in the end points of the two types of cones being $\pm$ 3°C.

Future work : (i) The following Orton type cones will be developed on a laboratory scale: (a) Cone Nos. 022 to 011; 02 to 4

and 11 to 14. Cone Nos.14-42 will be developed after receipt of the suitable testing equipment, (b) Cone Nos. 010 to 4 using iron oxide and lead oxide; (ii) Cone Nos. 022 to 14 are proposed to be made on a pilot plant scale with a view to standardise the production process.

Survey and market data on pyrometric cones:

A survey was undertaken in order to assess the annual requirements of pyrometric cones in India by the different consuming industries like the ceramics, iron and steel, etc.

While the central sources could provide information on the manufacturing units in India, it was not possible to estimate the potential requirements. Import figures were also not available. Only two firms, namely (i) Messrs Ganga Glass Works Private Ltd., Balawali Dist. (U.P.), and (ii) Messrs Bombay Potteries Tiles Ltd., Kurla, Bombay, were reported to be producing pyrometric cones. They were contacted and information furnished by them is given below.

Name of the firm	*Information supplied*
(1) Messrs. Ganga Glass Works, Private Ltd., Balawali Dist. (U.P.)	The cones are prepared according to the Staffordshire standard for the last six years and the raw materials used are indigenous.
(2) Messrs. Bombay Potteries Tiles Ltd., Kurla, Bombay.	*Annual requirements:* 4,380 dozens. *Annual production:* 5,000 dozens. *Temperature range of the cones*: 790° to 1350°C. *Price*: Rs. 25/100

Messrs. Mysore Iron and Steel Works, Bhadravati, have provided the following data:

Annual requirements:	1. The steel foundry	192	dozens
	2. Refractories Department	83.33	dozens

It is proposed to initiate a survey to assess the annual requirements of pyrometric cones in India by using statistical techniques.

38. REFRACTORIES

Synthesis of silicon carbide

Object: To synthesise silicon carbide grains required for the refractory and abrasive industries using locally available quartz and to study the properties of the silicon carbide.

Introduction: Silicon carbide (grain) is a totally imported item at present. The grain as well as finished products fabricated therefrom find a wide variety of uses in the ceramic and metallurgical industries and in thermal and nuclear engineering. The diversity of the application of silicon carbide products is based on the wide range in the physical, chemical, thermal and electrical properties which may be achieved by suitable variation of the composition and the conditions of synthesis of the grain as well as on the method of fabricating the products.

The synthesis of silicon carbide will, in the initial stage, relate to the determination of the suitability of main raw materials required for the purpose, a study of the conditions which affect the yields, and a study of the kinetics of the overall reaction.

Experimental work: The main equipment required for the synthesis of silicon carbide, i.e., a high temperature carbon resistor type furnace capable of being operated at temperatures up to 3000° C. has been installed. An auxiliary Scott's type transformer has also been included in the equipment. A suitable hood for the exit of hot gases from the furnace has been constructed, and an arrangement for the creation of reducing atmosphere at the entry and exit ends of the muffle furnace has been devised.

Future work: (i) A suitable method will be devised for the identification of silicon carbide and its quantitative estimation in the reacted product containing (a) silicon carbide, (b) various forms of unreacted silica, (c) various forms of unreacted carbon, and (d) silicon. X-ray diffraction, chemical analysis, physico-chemical methods like heavy media separation and flotation methods will be used.

(ii) The conditions for obtaining the optimum yields of *B*-silicon carbide, will be studied by using the following material:

Source of silicon:
1. Andhra quartz; trydimite and crystobalite prepared from Andhra quartz will also be used.
2. Silicon metal (for the purpose of comparison of the rate of reaction)

Source of carbon:
1. Petroleum coke
2. Coconut shell carbon
3. Coke obtained by carbonising pitch which will be prepared from L.T.C. tar
4. Graphite and retort carbon.

(iii) The effect of the composition, the time and temperature of the reaction, addition of minor additives on the conversion of *B*-Si C to α-Si C will be studied.

(iv) Particle shape and size, optical and X-ray properties and crystal growth of *B*-and α-Si C synthesized during the course of the work will be studied.

X-ray, Physico-Chemical studies & Instrumentation

39. X-RAY STUDIES

X-ray studies of coals and some other carbonaceous materials

Object : A systematic study of mineral matter and carbon content in coals, lignites and other carbonaceous materials employing the X-ray methods, and determination of the structural changes taking place on carbonisation and activation under different conditions.

Experimental work and conclusions: (See ACTIVE CARBONS under *Heavy Chemicals & Fertilizers.*)

40. PHYSICO-CHEMICAL STUDIES

Object : (i) To assist the research workers in various projects in physico-chemical studies.

Work during the year : The details of the project work have been reported under the respective research projects. The physico-chemical studies undertaken and equipment employed are cross-referenced below:

Project	*Equipment Employed*	*No. Of Samples Examined*
1. FATTY ACIDS	DU Spectrophotometer	41
2. DEHYDRATED CASTOR OIL (D.C.O.)	do	5
3. POTENTIAL PHARMACOLOGICAL COMPOUNDS (Carbostyrils and dihydrocarbostyrils)	do	24
4. CELL CONCENTRATION EFFECT	do	790
5. PROTEIN & NUCLEIC ACID BIOSYNTHESIS AND METABOLISM	do	14
6. WHITE CEMENT 7. CERAMICS & GLASS DEVELOPMENT PROJECT (Porcelain and enamels)	Reflectance spectrophotometer	7
8. CYMBOPOGON MARTINI (PALMAROSA & GINGER GRASS) OILS	Polarimeter	5
9. UTILISATION OF PRODUCTS OF L.T.C OF COAL (Chemical Examination of tar acids.)	Vapour phase chromatography (V.P.C.) apparatus	30
10. UTILISATION OF TURPENTINE	do	—

Microphotometer: Owing to the non-availability of the petrix batteries for getting their constant voltages, viz., + 100, - 80 V. to feed to the lamp source, voltages were obtained using wire ohm potentiometers in a circuit designed in the Laboratory. Microphotometer has been set up and is being used to find out the intensities of X-ray diffraction lines in order to estimate quantitatively the percentage of the quartz present in coal, coke and active carbons.

41. INSTRUMENTATION SERVICE

Object: (i) To maintain, service and repair the instruments in use in the Laboratory, (ii) to test and instal new instruments received, and (iii) to design and fabricate instruments to meet the requirements of research workers.

Work during the year:

(i) *Maintenance*: Approximately two hundred work orders were executed. These covered repairs and maintenance of instruments such as ultraviolet lamps, pressure gauges, transformer of Leitz heating microscopes, Cenco Hyvac pump, mercury switch of autoclave, pyrometers, Beckman pH meter model H-2, millivolt pyrometer H&B, Browns' temperature indicator, humidity chamber, magnetic stirrers of autoclaves, super centrifuge, calculating machines, etc.

(ii) *Test & Installation*: One 3-point temperature recorder and six indicators were installed on the control panel of the tar distillation pilot plant.

(c) *Fabrication*: (i) A heater was made on a glass condenser and a thermocouple was fabricated to measure the temperature: (ii) A 10-pin switch was made to measure the temperature.

Analytical

42. ANALYTICAL SERVICE

Object: To provide analytical services to government departments and industries, as also technical advice based on analytical studies.

Previous work : Analysis of samples of water, gulmohua flowers, soaps, inks, varnishes, polishes, chemicals, clays, and minerals was undertaken and technical advice given (Annual Report, 1957-58, p.92).

Work during the year :

General analytical work to assist Government departments and the private industries was continued. Nearly one hundred samples comprising chemicals, clays, cottonseeds, fertilizers, inks, ores and minerals, pharmaceuticals and water were analysed. Of these, thirty samples were received from government departments and the rest from the industries.

A micro-analysis laboratory to assist the research divisions is being installed.

Chemical Engineering

43. VAPOUR-LIQUID EQUILIBRIUM STUDIES

Object: Determination of vapour-liquid equilibrium data of fatty acids and tar acids to be used for design purposes.

Experimental work and conclusions: A new type of vapour-liquid equilibrium still has been designed and fabricated. The design is based on the modification of Gillespie type of still having a Cottrell pump.

In order to test the still for entrainment losses, a 5 per cent solution of sodium chloride was charged to the still and boiled for about four hours. At the end of this period the liquid and vapour samples were analysed and found to contain only about 0. 05 per cent, which is negligible.

In order to test the still for the reproducibility of the results, vapour-liquid equilibria of ethanol-water at atmospheric pressure were collected. Using Van Lar Equation, the vapour-liquid equilibria at 760 mm. were estimated. The results agree with the standard data.

Future work: (i) The still will be used for the determination of vapour-liquid equilibrium data of ethanol-water mixtures at about 50 mm. to standardise the conditions ;

(ii) Study will be made on vapour-liquid equilibria of (a) binary mixtures of saturated fatty acids under vacuum, (b) binary mixtures of saturated and unsaturated fatty acids, (c) ternary mixtures of fatty acids, and (d) binary mixtures of tar acids under vacuum.

44. VAPOUR PHASE ESTERIFICATION

Object: To study the esterification of butanol and acetic acid in vapour phase and study the kinetics of the process.

Work during the year: Literature review was made and the programme of work chalked out. The apparatus required was designed, fabricated, istalled and tested. The rate of flow of alcohol and acetic acid did not remain constant and various methods were tried to check the variations. Preliminary work is being done with ethanol and acetic acid to standardise the apparatus. Attempts are being made to procure micro feed pumps for alcohol and acetic acid.

Future work: After the preliminary work with ethyl alcohol and acetic acid to standardise the apparatus and experimental technique, it is proposed to study the kinetics of esterification of butyl alcohol and acetic acid with a number of catalysts at various temperatures and pressures with several molar ratios of alcohol and acid.

45. ADSORBENTS AND CATALYSTS

Studies on surface properties of adsorbents and catalysts.

Previous work: Literature survey was made and programme of work was chalked out. A volumenometer for the measurement of true density was fabricated and some preliminary work was done.

Experimental work and conclusions: (See ACTIVE CARBONS under *Heavy Chemicals and Fertilizers.*)

Future work: Porosity studies will be made using helium. A simple routine apparatus for surface area measurement based on the B. E. T. principle will be fabricated and used for these studies. For surface area measurement a continuous method will also be used based on the B. E. T. principle making use of a stream of mixtures of helium and nitrogen.

COLLABORATIVE PILOT PLANT WORK:

1. Plant for production of chemical cotton from linters

The design of a plant for three tons per batch capacity for the production of chemical cotton from cotton linters was prepared at the request of a private party. On the basis of the process details worked out, the digestion of three tons of cotton linters in sixteen hours was sufficient to get an output of four tons of chemical cotton in one working day of twenty four hours. The design of the plant was therefore based on the digestion capacity of three tons of linters per sixteen hours.

The qualitative flow sheet, material flow diagram for the process, sketches and blue prints of the units were prepared. The design of the plant essentially consisted of the following units: (i) Digester for a digestion capacity of three tons of linters, (ii) two pre-chlor beaters, capable of handling 500 lbs. per batch at a pulp cosistency of 3 per cent, (iii) two bleachers, necessary to keep the process continuous, (iv) post-chlor beater similar to (ii) above, and (v) Dryer with eight separate heater zones, capable of drying about 10,800 lbs. of cellulose at controlled moisture conditions.

2. Levulinic Acid: (See *LEVULINIC ACID* under *Organic Chemistry, Drugs & Pharmaceuticals.*)

3. Chlorination of turpentine: (See *PESTICIDES* under *Essential Oils and Aromatic Chemicals.*)

4. Hykol 'O' (Active Carbon Grade I): (See *ACTIVE CARBONS* under *Heavy Chemicals and Fertilizers.*)

5. Hykol 'X' (Active Carbon Grade II): (See *ACTIVE CARBONS* under *Heavy Chemicals and Fertilizers.*)

6. Bleaching Earths: A report on the requirements and conditions of processing 5 tons/day of fuller's earths has been prepared and sent to the Rajasthan Government. The report covers the following details: (1) Detailed description of the process of activation, (2) details of equipment for control and testing of raw materials and products at various stages, (3) features on which the

design of equipment for the project has been based and the necessary estimates, (4) flow sheet of the process with material balance at each stage, (5) description of operation, (6) staff requirements, (7) requirements of utilities, (8) estimated cost of production, and (9) sketches for different units of the plant.

7. Fertilizers : (See *FERTILIZERS* under *Heavy Chemicals and Fertilizers.*)

Operational Research, Technical Information and Industrial Liaison

46. OPERATIONAL RESEARCH

46. 1. Operational Research Unit

Object: To plan, coordinate and periodically evaluate the research undertaken in the Laboratory.

Introduction: The methods of operational research have been applied to the organisation and conduct of research projects in this Laboratory since 1949. This field of science helps in assessing the possibilities of an industrial problem even before it is actually taken up for investigation. The methods of operational research in this Laboratory include (a) the study of the past work on the research problem, (b) plan of research, (c) its place in the pattern of work of the Laboratory and (d) the scope of operation on pilot plant scale and eventually on an industrial scale. Likely impediments by way of availability of the raw materials, costs, social and economic factors, etc., are studied in advance and measures to overcome them are considered. Parallel schemes comprising laboratory research on the problem, collecting the pilot plant specification, raw material costs and market data, surveys through questionnaires and by personal contacts start functioning simultaneously. The operational research unit keeps the overall picture in continuous review and co-ordinates the functioning of the parallel scheme as a planned project.

The Operational Research Unit comprises a team of the research workers, chemical engineers, operationl research specialists and the Director. The Unit in its periodic meetings evaluates the

progress of work and plans coordination of the work on problems coming under the purview of specific disciplines.

When the technical and economic feasibility of a process has been ascertained through laboratory and pilot plant studies, a Non-Technical Note is drawn up for presenting the details of the process to the Industry. Interested parties are assisted in further pilot plant trials, training of technical personnel and, if required, in design, procurement and installation of equipment.

The laboratory facilities and research experience are thus placed at the service of the country, by advising the government and industry through project reports, project cost studies, etc., for establishing new industries in the country.

Work during the year: Twenty five meetings of Operational Research Unit (O.R.U.) were held to review the progress of various research schemes and projects under investigation in the Laboratory.

These O. R. U. meetings were further supplemented by group discussions generally attended only by workers in a particular discipline who go into the technical details of the problem and study the further course of work for the smooth progress of the research problems.

Future work: The work will be continued and developed as outlined above.

46. 2. Industrial surveys, market data

Object: (i) To assess the economic feasibility of various projects or schemes at the pre-investigation and pilot plant investigation stage on the basis of the statistical and market data available; (ii) when a project relates to the development of a process for the production of marketable products, survey is carried out to assess the existing and potential market for the products.

Previous work: The surveys that have been undertaken cover the following projects and schemes: wax-like products from castor oil; fatty acids; problems of the paints industry; citric, levulinic and itaconic acids and their salts; hand-made paper; products and by-products from low-temperature carbonisation of coal; active

carbons; hydrazine sulphate and hydrate; sodium sulphide; glue; problems of the glass and ceramic industry; and standard pyrometric cones.

Work during the year:

A. SURVEY AND MARKET DATA:

Work on surveys and collection of market data on different projects has already been described under the respective projects and a cross-reference is made in this chapter.

(1) *Fatty acids*: (See FATTY ACIDS under *FATS & OILS)*

(2) *Cashewnut shell liquid*: (See CASHEWNUT SHELL LIQUID under *SURFACE COATINGS*).

(3) *Products of low-temperature carbonisation of coal*: (See under *FUEL*).

(4) *Fuller's earth and bentonitic clays*: (See BLEACHING EARTHS under *HEAVY CHEMICALS & FERTILIZERS*).

(5) *Standard pyrometric cones*: (See under CERAMICS).

Further information on these surveys not given under the projects is detailed here.

(6) Active Carbons:

A mail questionnaire survey was initiated to assess the annual requirement of different consuming industries such as vegetable oils, sugar, glycerine, chemicals and pharmaceuticals. Questionnaires were sent to all the vanaspati ghee (hydrogenated oil) manufacturers in the country. A 25 per cent sample of the chemical and pharmaceutical concerns was also chosen from the *Thacker's Indian Directory (1956–57)* and these were addressed to furnish the requisite information. The survey will be extended to other industries mentioned above.

(7) White cement:

(a) *Annual requirements*: The exact requirement figures were not available. However, on the basis of the imports and production

figures available, the requirement of white cement is estimated as 7,000 tons per annum.

(b) *Indigenous production*: Messrs. Associated Cement Co., Ltd., have reported an annual production of 2,500 tons of white cement and plans to manufacture about 5,000 to 6,000 tons. The product manufactured by the above firm is marketed under the name of 'Silvi-crete' at Rs. 276 per ton.

(c) The c. i. f. price of imported white cement varied from Rs. 250 to Rs. 310 per ton during 1957.

(8) Petroleum coke and electrode carbon:

(a) *Indigenous production:* Only the Assam Oil Company in the public sector produces petroleum coke in India. The production in 1957 was 12,445 tons (communication from the Ministry of Steel, Mines & Fuel) and there is a proposal to raise the production to 39,000 tons (communication from the Planning Commission).

(b) *Annual requirements of the aluminium industry*: The aluminium industry is the major consumer of petroleum coke. The consumption of Soderberg electrodes by the two major aluminium concerns is as follows:

Name of the factory	Consumption (tons)						
	1951	1952	1953	1954	1955	1956	1957
1. M/s. Indian Aluminium Co., Ltd., Calcutta.	1,380	1,587	1,308	1,959	3,211	2,613	3,356
2. M/s. Aluminium Corporation of India, Ltd., Calcutta.	7760 (for the entire period)						

The second reduction works at Hirakaud of Messrs. Indian Aluminium Co., Ltd., are expected to consume 6,000 tons of Soderberg electrodes in the initial stage (10,000 tons per annum of aluminium ingot production capacity) and 12,000 tons when the production capacity is expanded (20,000 tons ingots per annum).

On the basis of the above data, it was estimated that the annual requirement of petroleum coke by the aluminium industry was about 2,600 tons. It was estimated that requirements would increase to 10,000 tons by the end of 1961.

The prices of the imported coke were U. S. A. $68.42 per long ton and that of the indigenous coke Rs. 166.32 per ton. The specifications of both the cokes have been obtained.

(9) Gypsum and lignite :

Data was collected on (i) the place of occurrence, (ii) the extent of deposits, and (iii) estimated reserves.

GYPSUM

(i) *Occurrence* :

Place of occurrence	Estimated reserves	
	Geological Survey of India	Fertilizer Production Committee
RAJASTHAN :		
Bikaner	28,241,000	125,000,000
Jodhpur	21,320,766	Of this quantity Jamsar in Bikaner alone contains 30 million tons.
Jaisalmer	1,341,000	
	50,902,766	
MADRAS :		
Trichinopoly	15,300,000	30 million tons.
Nellore	1,000,000	
	16,300,000	
SAURASHTRA :		
Halar Dist.	4,441,000	
Bhavanagar, Porbandar	25,000	
	4,466,000	

KUTCH:	2,071,000
HIMACHAL PRADESH:	
Sirmur	383,000
UTTAR PRADESH:	
Lachmangula	156 000
Dehra Dun	13,000
Nainital	37,000
	206,000

(ii) *Production*:

State	Production (tons) 1953	1954	1955	1956
RAJASTHAN:				
Bikaner	357,381	320,815	390,377	494,066
Jodhpur	188,164	224,254	77,861	74,640
Barner	—	--	169,458	233,261
MADRAS:				
Tirichinopoly	34,992	58,529	39,697	27,105
Tirunelveli	—	--	4,413	3,013
Ramnathpura	—	1,439	2,029	97
Coimbatore	—	2,017	5,003	17,401
SAURASHTRA;	4,607	1,404	—	—
KUTCH:				
Lakhpet	690	200	—	--
UTTAR PRADESH:				
Garhwal	--	3,639	1,067	—
KASHMIR:	5	23	—	—

LIGNITES

Place of occurrence	Estimated reserves (tons)	Remarks
MADRAS:		
South Arcot Dist.	2,000 million; (of these the more workable deposits amount to 200 million)	*Chemical analysis*: fixed carbon 22%, volatiles 25%, ash 3%, moisture 50%. *Heating value*: about 5,500 B.T.U./lb., (about 1/3 of the high grade coal).
KASHMIR:		
Shaliganga to Ferozpurnala	37.3 million	
Tangmarg	6.3 ,,	
Naghal Baramulla	11.1 ,,	
Baramulla	7.4 ,,	
Nichoma	13.8 ,,	
Chowkibal	7.5 ,,	
BOMBAY:		
Umarsar	4.0—4.3 million	*Chemical analysis*: (%) moisture 21.28, 21.50, 19.50; volatiles 42.54, 28.98, 47.30; fixed carbon 25.64, 17.32, 32.40; ash 6.54, 32.40, 10.12; *fuel ratio*: 0.60, 0.60, 0.60
Lofri	134,000 ,,	
Thulari	1,400,000 ,,	

46. 3. INDUSTRIAL LIAISON

A. Visits to Industries :

Visits were arranged by teams consisting of the research workers and members of the Operational Research, Technical Information and Liaison Division to the industries to acquaint themselves with the problems and outlook of the industry. The details are given below :

Name and address of industry	*Purpose of visit*
1. Associated Cement Co, Ltd., Shahabad.	Study of the process and plant for manufacture of cement on industrial scale.
2. Hyderabad Potteries Ltd., Hyderadad. 3. Charminar Potteries Ltd., Secunderabad. 4. Hyderabad Laminated Products Ltd., Hyderabad.	Ceramic survey, study of manufacturing processes and instruments for testing electrical properties of ceramic materials.
5. Sirpur Paper Mills Ltd., Sirpur-Kaghaznagar. 6. Sirsilk Ltd., Sirpur, Kaghaznagar.	Study of processes and the industrial plant operations.
7. Swastik Bobbins Manufacturing Co., Pr. Ltd., Azamabad, Hyderabad.	Technical advice on the utilisation of sediment collecting in French blue enamels.
8. Aryan Industries Pr. Ltd., Secunderabad.	General information.
9. Raj Mineral Works, Hyderabad.	Filtrability tests of different qualities of sand.
10. Kapadia Oil Mills, Azamabad, Hyderabad.	General Information.
11. Snow White Food Products Co. Ltd., Howrah.	General Information.
12. Hindustan Insecticides Pr. Ltd., New Delhi.	Study of the chlorination plant.
13. The Anjerakandi Oil Co. and the Brown Cinnamon Estate, Kerala.	Survey of cinnamon leaf oil production.

B. Visits to Research Centres and Government Departments

	Name and address		*Purpose of visit*
1.	Engineering Research Laboratories, Hyderabad.	...	Equipment for cement testing according to ISS and other testing facilities.
2.	Department of Mines and Geology, Andhra Pradesh, Hyderabad.	...	Information about availability of the barytes ore in the State.
3.	Department of Chemical Technology, Osmania University, Hyderabad Dn.	...	Information on equipment and testing facilities.
4,	Radium Institute and the Cancer Hospital, Hyderabad.	...	Equipment for measuring dosages of gamma rays for therapy and self-protection.
5.	Central Glass and Ceramic Research Institute, Calcutta.	...	To study production of pulverised mica and the manufacture of optical glass.
6.	Government Test House, Alipore, Calcutta.	...	Paint Testing Section
7.	Shri Ram Institute for Industrial Research, Delhi.	...	Viscose spinning pilot plant and general information on research projects.
8.	Central Road Research Institute, New Delhi.	...	Tests on the suitability of low temperature tar as a road tar.
9.	National Physical Laboratory, New Delhi.	...	Study of pilot plants and general information about research programmes.
10.	Indian Agricultural Research Institute, New Delhi.	...	General Information about research programmes.
11.	The Government Industrial Laboratory, Trivandrum (Kerala).	...	Survey of cinnamon leaf oil and products.

C. Visits Abroad

	Name	*Places visited*
1.	Dr. S. Husain Zaheer (as a member of the Indian Delegation)	Paris: to attend the General Conference of UNESCO, Nov. 4-Dec. 4, 1958.

Name	*Places visited*
2. Dr. P. M. Bhargava (at the invitation of the International Congress of Biochemistry).	1. Vienna: to attend the Fourth International Congress of Biochemistry.
	2. Munich: Max Plank Institute for Biochemistry.
	3. Paris: Pasteur Institute.
	4. Brussels: University of Brussels.
	5. London: National Institute for Medical Research.
	6. Amsterdam: University of Amsterdam (Prof. Slater's Laboratory); and the Cancer Institute.
	7. Heidelberg: Institute for High Polymers Research.

46. 4. Literature notes and schemes

Object: To compile literature background of previous work oned and a plan for the proposed scheme of research, with an indication of its industrial setting in the country. This is enable consideration and decision for its being put on laboratory research programme by the Operational Research Unit and the Director.

Introduction: Literature notes and schemes of research in general precede the taking up of a research problem, to justify the proposal resting on a sound basis. Some schemes are also put up to various financing bodies and the State governments indicating specifically the necessity of undertaking the problem and the extent of financial help required.

The literature notes outline the previous work on the subject, rationalise the programme of research proposed, indicate relevant statistical and economic data and indicate the basic objective and time target.

The titles of the literature notes etc., prepared during the year:

1. Cinnamon leaf oil.
2. Catalysts for hydrogenation of low-temperature tar.
3. Heat of wetting and surface area of coal.
4. Production of sodium on a small scale.
5. Preparation of standard pyrometric cones.
6. Preparation of pyrometric sheaths from indigenous materials.
7. Alkyds.
8. Polyhydroxy compounds as film formers.
9. Use of aluminium powder pigment in primers and its comparative evaluation to conventional primer pigments.
10. Optical bleaching agents in paper industry.
11. Utilisation of paper mill waste in India.
12. Chemicals for increasing the wet strength of paper.
13. Economic bleaching of fast coloured rags, (cotton).
14. De-inking of waste papers.
15. Conversion of pinenes to terpeniol.
16. Camphor from pinenes.
17. Physico-chemical studies on active carbons, Part I. Methods described in literature for measurement of surface area, pore volume and pore size distribution in active carbons.
18. Physico-chemical studies on active carbons, Part II. Calculation of surface area, pore volume and pore size distribution of active carbons prepared in the laboratory

using data on adsorption of methylene blue and benzene previously determined.

19. Nature of work that can be undertaken using Nernst Tammann Furnace.

20. Living insecticides.

46. 5. Non-technical notes and reports

Object : (i) With a view to interest industry, government departments and other interested parties, non-technical notes and project costs on completed or nearly-completed laboratory processes or projects are compiled.

(ii) Detailed project reports embodying the technical, statistical and the estimated cost data are prepared to assist industries in the public and private sectors.

Work during the year :

The titles of the non-technical notes and the projects prepared during the year are given below :

(i) CITICIDE

(a) for National Research Development Corporation of India, New Delhi.

(b) for Messrs. Tata Fison Private Ltd., Bombay.

(2) Indian bentonites.

(3) Non-caking ammonium nitrate fertilizer.

(4) Utilisation of iron pyrites.

(5) Sulphur from gypsum.

(6) Preparation of hydrazine sulphate.

(7) White cement from felspar.

(8) Bleaching of mineral oils.

(9) Reclamation of used lubricating oil.

(10) Sutlej river silt as a diluent and coating agent for ammonium nitrate fertilizer.

(11) Pre-design cost estimates on the manufacture of sodium sulphide.

(12) Pre-design cost estimates for the production of white cement from felspar.

47. TECHNICAL ASSISTANCE, AND ENQUIRIES

47. 1. Technical assistance

Object: To place the experimental facilities of the Laboratory and its experience at the service of industry.

Work during the year:

(i) *Preparation of purified linoleic acid and linolenic acid:* 3 gms. samples of each of the above chemicals were prepared and sent on request to Dr. Ganguly, Indian Institute of science, Bangalore, for spectrophotometric standardisation purpose.

(ii) *Supply of refined cottonseed oil.* 2 lbs. of cottonseed oil was refined and sent to Indian Central Oilseeds Committee.

(iii) *Reclamation of contaminated insect repellant.* A method was worked out for reclamation of contaminated repellant after a careful study of the stock sent by the Defence Ministry.

(iv) *Grammage test of carbon paper samples.* Grammage tests of samples of carbon paper received from the Director, Printing and Stationery, Andhra Pradesh, were carried out and a test report was sent.

(v) *Determination of the P.C.E. value of fire clays and fire brick samples.*

The P.C.E. value of fire clays and fire brick samples received from Messrs. Jawahar Industries, were determined and a note was sent.

(vi) *Utilisation of sediment collecting in the French blue enamel.*

A sample in which the sediment had been effectively incorporated into the vehicle were prepared and sent to Messrs. Swastik Bobbins Mfg. Co. Ltd., Azamabad, and technical know-how was provided.

(vii) *Chemicals and equipment required for the production of Lounginin.*

The quantity of the chemicals required for the annual production of 2000 lbs. of Lounginin and the list of equipment required were prepared and sent to Messrs. Biological Products, Pvt. Ltd. Hyderabad.

(viii) *Calendering of demi size paper.*

2000 sheets of demi-size paper prepared by a local paper manufacturer were calendered to obtain proper finish required by the party.

(ix) *Training of artisans.* Under a scheme sponsored by the Khadi and Village Industries Commission, Bombay, on the production of high grade papers from rags, training course was given to five citizens and certificates awarded after completion of the course.

(x) *Chlorophyll estimations*: 30 chlorophyll estimations were done for research workers from the Department of Botany, Osmania University.

(xi) *Plasticity number and sieve analysis of pencil clays.*

The plasticity number and sieve analysis of pencil clays received from M/S. The Quilon Pencil Factory were carried out.

(xii) *Technical know-how for setting up of a plant for manufacture of chemical cotton from second-cut cotton linters.*

The necessary technical assistance and advice is being offered to M/s. Bhagubhai Chandulal, Ahmedabad, for setting up of a plant for the manufacture of chemical cotton.

(xiii) *Processing of cottonseed.* The processing of cottonseed was carried out at the pilot plant for M/s. Kapadia Oil Mill, Hyderabad.

(xiv) *Maintenance of uniform heat in the heating chamber for enamel drying.*

The heating chamber of M/s. Swastik Bobbins Manufacturing Co., (Pr.) Ltd., was inspected by an officer from the Laboratory and the necessary assistance was given.

(xv) *Testing of sample of stationery sealing wax*

The testing was carried out and a report sent to the Director of Stationery, Andhra Pradesh.

47. 2. Technical enquiries

	Information on	*Enquiry from*
1.	Estimated production ; specifications and cost of production and refining cotton linters.	Shri Ram Institure for Industrial Research, Delhi.
2.	Percentage of Paper shellac required in the varnish for paper varnish.	Central Stores Purchase Dept., Andhra Pradesh.
3.	Corrosion problems in chemical industry.	Regional Liaison Officer, Calcutta.
4.	Equipment for testing P. C. E. refractories under load and other tests.	The Mysore Iron and Steel, Works, Bhadravati.
5.	Fattening tendency in the red lead ready-mixed paint.	Associated Cements Ltd., Calcutta.
6.	Spontaneous combustion of coal during storage.	Regional Liaison Officer, Madras.
7.	(i) Modification of the process of open-to-air cooking at high temperatures with the vitaminised cooking medium to retain vitamin A.	The United Vegetable Manufacturers Ltd., Calcutta.

	(ii) Any method developed to avoid the loss of seasonal contact during the post-refining stage when neutralised and bleached sesame oil is added.	Manufacturers, Limited.
8.	The kind of packing required to preserve herbal leaves.	P. N. K. Subbaram, Madura.
9.	List of the names and addresses of essential oil dealers in India.	Fratalli Caminiti, Italy.
10.	Preparation of fatty amines and methyl ester of castor oil.	Mazda Trading Syndicate, Bombay.
11.	List of the names and addresses of cottonseed oil mills in Andhra Pradesh.	Shri S. A. Samual Roy, Vellore.
12.	Appropriate metal for construction or lining etc., for reactor vessels required in $AlCl_3$ manufacture.	Regional Liaison Officer, C. S. I. R., Bombay.
13.	Substitutes for raw materials for (i) pyridine, (ii) essences and (iii) glues.	The Secretary, C.S.I.R. New Delhi.
14.	Substitutes for chlorinated naphthalene used in the flame-proofing treatment.	Devendra Brothers, Kanpur.
15.	Extraction of croton oil.	Dr. Y.H. Daftary, Bombay, 18.
16.	Denaturant grade wood naptha.	The Director of Industries & Commerce, Andhra Pradesh.

17.	Suitable lacquers for varnishing artificial silk braided electrical cable.	B. D. Gopal Dass, Kanpur.
18.	Technical definition of crude and refined saltpetre.	The Singareni Collieries Co. Ltd., Andhra Pradesh.
19.	Possible uses of sandstone, red oxide and iron oxide.	Harmukhlal Rugnath Shah & Co., Bombay.
20.	Removal of smell and dust from fats and oils.	Shri Maqbool Ali, Gulbarga.
21.	Possibility of using lignite for the burning of limestone and replacing coke by lignite in the final conversion of lime to calcium carbide.	Regional Liaison Officer, C. S. I. R. Madras.
22.	Paints and varnishes required for fabric-cum-metallic tapes.	The Ludhiana Measuring Tape Co., Ludhiana.
23.	Manufacture of silica gel.	The National Chemical Industries, New Delhi.
24.	Blackboard paints.	Head Master, C. A. V. High School, Hissar.
25.	By-products of molasses.	Kapadia Industries, Hyderabad.
26.	Analytical data in respect of groundnut oil cake for framing specifications.	Agricultural Marketing Adviser, Nagpur.
27.	Purification of carbon-dioxide during the process of fermentation.	Regional Liaison Officer, C. S. I. R. Madras.
28.	Colour removal and the reduction of acidity in the spirit after distillation.	-do-

29.	Equipment for the production of hydrogen gas.	Sarabhai Chemicals, Baroda.
30.	Preservation of vegetable oils.	The Secretary, I. C. O. C. Hyderabad.
31.	Comparative uses of pure alcohol and pure benzene in a solvent extraction plant for extracting oils from vegetable oilseeds and cake.	Regional Liaison Officer. C.S.I.C. Madras.

Enquiries pertaining to the Laboratory and its processes:

Information on	*No. of enquiries*
Cottonseed	13
Fatty acids	4
Surface coatings	1
Palmarosa oil	2
Dehydrated castor oil	5
Laboratory	2
Benzyl chloride	1
White cement	1
Terpeniol	2
Hand-made paper	1
Lounginin	2
Citicide	14

PUBLICATIONS AND PATENTS

Research papers published :

1. Studies on indigenous cottonseed, Part II. Storage of cottonseed, V. R. Harwalkar, V. V. R. Subrahmanyam and S. A. Saletore, *Indian J. Appl. Chem.*, 1958, *21*, 66-68.

2. Studies on indigenous cottonseed, Part III. Storage and stability of cottonseed oil, V.R. Harwalkar and S.A. Saletore, *ibid.*, 69-73.

3. Studies on indigenous cottonseed, Part V. Delinting, K. S. Chari, V. R. Harwalkar, G. S. Shenolikar, S. Zainulabideen, S. A. Saletore and S. Husain Zaheer, *J. Sci. Industr. Res. (India)*, 1958, *17A*, 371-75.

4. Storage of cottonseed, V. R. Harwalkar, S. A. Saletore and S. Husain Zaheer, *Proc. Fourth Symp. on Oils, Fats and Allied Products (Bombay)*, 1955, publ. 1958, pp. 7-10.

5. Pretreatment and processing of cottonseed, V. R. Harwalkar, K. S. Chari, S. A. Saletore, and S. Husain Zaheer, *ibid.*, pp. 101-105.

6. The effect of alkali excess on the refining of cottonseed oil, V. P. Harigopal, S. Raghavendar Rao and S. A. Saletore, *ibid.*, pp. 129-32.

7. Hyderabad earths for bleaching vegetable oils, S. S. Joshi and S. A. Saletore, *ibid.*, pp. 138-39.

8. Bleaching earths for vegetable oil refining, S. S. Joshi, M. A. Hai and S. A. Saletore, *Paintindia*, 1958, *8*, 53-54.

9. Studies on indigenous cottonseed, Part VI - Delinting and hulling, K. S. Chari, V. R. Harwalkar, S. A. Saletore and S. Husain Zaheer, *J. Sci. Industr. Res. (India)*, 1959, *18A*, 66-68.

10. Crystallisation of Indian beef tallow fatty acids from aqueous ethanols, V. V. R. Subrahmanyam and K. T. Achaya, *J. Amer. Oil Chem. Soc.* 1958, *35*, 467-469.

11. Uber Tropische Pflanzen and Tierfetten, S. Husain Zaheer and K. T. Achaya, *Die Nahrung*, 1958, *2*, 710 - 18.

12. Coating compositions from modified kamala seed oil, M. C. Menon and J. S. Aggarwal, *J. Sci. Industr. Res. (India)*, 1958, *17B*, 279-81.

13. D. C. O. Varnishes and alkyds from castor oil, M. A. Sivasamban, S. Neelakant Rao, N. Bhojraj Naidu and S. A. Saletore, *Paintindia*, 1958, *8*, 47-50.

14. Chromatic identification of phenols in tar acids from low-temperature tar oils, Bharat Bhushan, Anant N. Dharmapuri, K. S. Chari, K. Ramachandran and S. H. Zaheer, *J. Sci. Industr. Res. (India)*, 1958, *17B*, 385-86.

15. Project costs, IV. CITICIDE, a new pesticide from turpentine, Bharat Bhushan, K. S. Chari, S. Jaleel Hasan, Naseemul Huq, C. C. Reddy and S. H. Zaheer, *Res. & Ind.*, 1958, *3*, 267-70.

16. On the mechanism of formation of itaconic acid by *Aspergillus terreus*, P. M. Bhargava, (late) K. Ramachandran, Shyamala Rao, Mohan Lal and S. H. Zaheer, *Suppl. Int'l. Abs. Biol. Sci. (IV International Congress of Biochemistry, Vienna)*, 1958, *Section 10*. Abs. No. 10-95, p. 136.

17. Briquetting of Hyderabad coals: Part III - Effect of moisture, mixing time and temperature, method of application of binder and particle size of coal on briquette strength, D. P. Agrawal, M. G. Krishna and S. H. Zaheer, *J. Sci. Industr. Res. (India)*, 1959, *18 B*, 33-38.

18. Vapour-liquid equilibrium relationships, C. C. Reddy and K. S. Chari, *ibid.*, 1958, *17 B*, 97-102.

19. Studies in lithium oxide systems : III - Liquid immiscibility in the system $Li0_2$ - B_20_3 - $Si0_2$, B. S. R. Sastry and F. A. Hummel, *J. Amer. Ceram. Soc.*, 1959, *42*, 81-88.

20. Solubilities of vegetable oils in ethanol and ethanol - hexane mixtures, Ramalingam Kaparthi and K. S. Chari, *J. Amer. Oil Chem. Soc.*, 1959, *36*, 77-80.

21. Laboratory investigations on the extraction of oil from vegetable oil cakes with ethanol, Ramalingam Kaparthi and K. S. Chari, *ibid.*, 1959, *36*, 81-83.

22. Inhibitory neurohumor of insects, M. B. Naidu, *Indian J. Entomol.*, 1958, *20*, Part II, 147-152.

23. La fabrication de gluconate de calcium par fermentation, S. Husain Zaheer, K. Ramachandran, C. C. Reddy and M. Zulfiqar Ahmed, *Produits Pharmaceutiques*, 1958, *13*, 201-203.

2. Semi-technical, review articles and notes published :

1. Standards for cottonseed and its products, K. T. Achaya, S. Raghavendar Rao, N. Sambasiva Rao and S. A. Saletore, *ISI Bull.* 1958, *10*, 118-21.

2. Chemistry and technology of castor oil, cake and stalks, K. T. Achaya, M. A. Sivasamban and S. Neelakant Rao, *Monograph on "CASTOR"* Indian Central Oilseeds Committee, Hyderabad, 1959, Chap. XI.

3. Cottonseed and its products - A symposium, *Res. & Ind.*, 1959, *4*, 53-55.

4. Refining of oils and fats, J. S. Aggarwal, *Indian Oilseeds J.*, 1958, *2*, 103-19.

5. Bhela Oil, J. S. Aggarwal, *J. Oil Col. Chem. Assocn.*, 1958, *41*, 684.

6. Kamala seed oil for paints and varnishes, J. S. Aggarwal *Paint Manuf.*, 1959, *29*, 50-52.

7. Coordinating dyes used as indicators in complexometry, N. Sambasiva Rao and G. S. Sidhu, *Hand Book of Symposium on Chemistry of Coordinating Compounds*, Agra, February 7 - 8, 1959, *Section VI*, p. 118.

3. Papers communicated for publication.

1. Preparation and utilisation of dehyrdated castor oil (D. C. O.), M.A. Sivasamban, S. Neelakant Rao, N. Bhojraj Naidu and S. A. Saletore, *Brochure, National Institute of Sciences (India).*

2. Mallotus philippenensis Muell. Ang. - Monkey face tree, J. S. Aggarwal. *Wealth of India* (Dictionary of Raw Materials).

3. Synthesis of some carbostyrils and hydrocarbostyrils, G. S. Sidhu, G. Thyagarajan and Miss Salma Ansari, *Annalen der chimie.*

4. The U. V. absorption spectra of some carbostyrils and hydrocarbostyrils, G. S. Sidhu, V. S. Subramanyam and K. M. Ghouse, *Annalen der chimie.*

5. The role of lipids in the susceptibility of an insect to insecticide, M. B. Naidu, *Indian J. Entomol.*

6. Felspar suitable for making white cement, Part I. Physical properties of Golconda felspar, E. R. Saxena, D. S. Datar and S. H. Zaheer, *Trans. Indian. Ceram. Soc.*

7. A note on Kerala ball clays, M. Noman Khan and Balabheem Rao, *Res. & Ind.*

8. Kerala ball clays, Part I. Physical properties of the raw clays, M. Noman Khan, B. Rama Rao and Balabheem Rao, *Trans. Indian Ceram. Soc.*

9. Fire clay refractories using some Andhra Pradesh and Mysore clay, M. A. Wahab and Balabheem Rao, *Trans. Indian Ceram. Soc.*

10. Pilot plant studies on low-temperature carbonisation of non-caking coals from Andhra Pradesh, S. H. Zaheer, M. G. Krishna, K. G. Rangrez and G. S. Chowdhury, (accepted for publication in *Indian Mining Journal*, presented at the *Symposium on 'Coal carbonisation'* held at Central Fuel Research Institute, Jealgora, March 19-24, 1957).

11. Processing of low temperature tar for road tar and pitch, B. S. Narayan Rao, R. Vaidyeswaran, Anant N. Dharmapuri, Mohd. Ehsan, M. G. Krishna and S. H. Zaheer. (accepted for publication in *Indian Mining Journal*, presented at the *Symposium on 'Coal carbonisation* held at Central Fuel Research Institute, Jealgora, March 19-24, 1957).

4. Papers read at symposia, conferences, etc.

I. Conference on 'High temperature Kinetics' Massachusetts Institute of Technology, U. S. A., June 1958.

1. Densification during sintering in the presence of liquid phase, Part II. Experimental, W. D. Kingery and M. D. Narasimhan.

II. Fourth International Congress of Biochemistry, Vienna, September, 1958.

1. On the mechanism of formation of itaconic acid by *Aspergillus terreus*, P. M. Bhargava, (late) K. Ramachandran, Shyamala Rao, Mohan Lal and S. H. Zaheer.

2. Incorporation of radioactive amino acid in bull spermatozoa, P. M. Bhargava.

III. Symposium on 'Utilisation oj vegetable oils and their products, National Institute of Sciences, New Delhi, October, 1958.

1. The separation of fatty acids using urea, K. T. Achaya and S. H. Zaheer.

IV. Symposium on 'Cottonseed and its by - products,' Regional Research Laboratory, Hyderabad, December 1958.

1. Proximate composition of some Hyderabad and Bombay cottonseeds, K. T. Achaya and S. A. Saletore.

2. Processing of cottonseed - delinting and dehulling, K. S. Chari, S. A. Saletore and S. H. Zaheer.

3. Storage of cottonseed and its oil, S. Raghavendar Rao, K. T. Achaya and V. V. R. Subrahmanyam.

4. Solubility of cottonseed oil in ethyl alcohol, K. S. Chari, K. Ramalingam and S. H. Zaheer.

5. Some laboratory studies on refining of cottonseed oil, V. P. Harigopal, S. Raghavendar Rao, K. T. Achaya and S. A. Saletore.

6. Hydrogenation of cottonseed oil, J. S. Aggarwal.

7. Utilisation of cottonseed oil refining foots, K. T. Achaya, Bharat Bhushan and S. Raghavendar Rao.

8. Studies on the preparation of chemical cotton, G. S. Sidhu, S. Zainul Abideen and Iqbal Ahmed.

9. Composition and urea separation of fatty acid from Indian cottonseed oil, K. T. Achaya and S. A. Saletore.

10. Standards for cottonseed and its products, Part II. Cotton linters and cottonseed cake, S. Raghavendar Rao and K. S. Chari.

V. First Conference of oilseeds research workers in India, Indian Central Oilseeds Committee (Hyderabad), Chandigarh, December 1958.

1. Fatty alcohols from castor oil and its esters Part I. Preparation by reduction and sulphation, V. V. R. Subrahmanyam and K. T. Achaya.

2. Emulsion varnishes based on dehydrated castor oil, M. A. Sivasamban and S. A. Saletore.

3. Kamala seed oil and its utilisation, J. S. Aggarwal.

VI. Symposium on 'Corrosion and its prevention', Society for Corrosion and Coating Technology, Calcutta, December 1951.

1. Anti-corrosive paints for ship bottoms, M. C. Menon and J. S. Aggarwal.

VII. Symposium on 'Electrolytic cells', Central Electrochemical Research Institute, Karaikudi, December 1958.

1. Co-deposition of cobalt nickel, V. S. Subrahmanyam.

VIII. Symposium on 'Proteins,' Society of Biological Chemists (India), Delhi, January 1959.

1. Mechanism of protein synthesis, P. M. Bhargava.

XI. Symposium on 'Clay minerals,' Chemistry Section, Indian Science Congress Association, Delhi, January 1959.

1. Fuller's earth deposits of Mysore State, S. S. Joshi and S. A. Saletore.

2. Mineral analysis of some Andhra Pradesh clays, A. V. Rajeswara Rao.

X. Symposium on 'Chemistry of co-ordinating compounds,' National Academy of Science (India), Allahabad, February 1959.

1. Co-ordination dyes used as indicators on complexometry, N. Sambasiva Rao and G. S. Sidhu.

XI. All India Paint Manufacturers Association, Kanpur, February 1959.

1. Drying oil resources of India, J. S. Aggarwal.

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Patent applications sealed :

1. Manufacture of 1-ethoxy-2-hydroxy - 4 - propenyl benzene, S. Mahboob, V. D. N. Sastri, K. Ramachandran and S. Husain Zaheer, Indian Patent Application No. 60863, 24th June 1957 (sealed on 11th February 1959).

2. A process for the separation of tallow and tallow-like fatty acids into mainly saturated and mainly oleic acid, S. Husain Zaheer, V. V. R. Subrahmanyam and K. T. Achaya, Indian Patent Application No. 62263, 12th November 1957 (sealed on 31st December 1958).

3. Improvements in or relating to vats for making hand-made paper, G. S. Chowdhury, M. K. Chary, S. A. Saletore and S. Husain Zaheer, Indian Patent Application No. 63289, 3rd March 1958 (sealed on 18th March 1959).

4. A process for the production of alkyl eugenols, S. Mahboob, C. C. Reddy and S. Husain Zaheer, Indian Patent Application No. 63719, 10th April 1958 (sealed on 11th March 1959).

Patent applications filed :

1. A process for the production of hydrogen sulphide from gypsum. Razia Osmani, D. S. Datar and S. Husain Zaheer. Indian Patent Application No. 65779, 12th November 1958.

2. Improvements in or relating to electrode holders from manual arc welding, G. S. Chowdhury, G. C. G. Reddi, M. K. Chary and S. Husain Zaheer, Indian Patent Application No. 66294.

General Engineering

Introduction: Pilot plant studies on processes completed on laboratory scale are essential for establishing its technical and commercial feasibility on an industrial scale. Design, fabrication and installation of pilot plants requires the assistance of a workshop. Selection of equipment, its testing, putting in operation and maintenance are also necessary.

For the units to be purchased, descriptive literature is invited from the supplying firms. Specifications are scrutinised by research workers, and the chemical and mechanical engineers before an order for purchase is placed.

Work during the year:

Engineering equipment or machinery purchased, and the items designed and fabricated in the Laboratory workshop are described.

1. Units procured, installed and under installation :

(i) Beche pneumatic hammer, (ii) Do-all machine, (iii) Ingersoll Rand air compressors, 5 nos., (iv) motor, 50 h. p. for ammonia

compressor, (v) "Chicago" Ducati intercomes, (vi) submersible pumps, 2 nos., (vii) oxygen plants from the Directorate General, Supplies and Disposals, 3 nos., (viii) 350-ton hydraulic drawing press from the DGSD, (ix) water coolers, (x) drying chamber in the Hand-made Paper and Cellulose Section, (xi) hand milling machine (xii) Tar distillation plant.

2. Design

Designs and drawings for the following units were made :

(i) Main structure for the fat splitting and fatty acid distillation pilot plant, (ii) structure and thrust bearing housing for the neutraliser, (iii) dust collector (bag type), (iv) ice and water cooling plant. (v) cyclone filter, (vi) gallery in the annexe between the pilot plant nos. 3 and 4, (vii) equipment and furniture for the (a) furnace room, and (b) cold room, (viii) fume cupboards and wardrobes for the L. T, C. laboratory, (ix) furniture for Post Office and gate lodge.

3. Units fabricated

(i) Dehydration tar still, (ii) apparatus for the breaking point of bitumen (Frauss method), (iii) fatty acid samplers of 10 cc. capacity and 1500 lb./sq.in. tested pressure, 2 nos., (iv) drum abrasion tester, (v) shell and tube cooler, (vi) steam jacketed Buchner funnel, (vii) wax lamps (four types), (viii) infrared heaters, 2 nos., (ix) wooden sand trap for Hand-made Paper and Cellulose Section, (x) drum screen for pulp beater, (xi) dies for making pyrometric cones, 2 sets, (xii) pattern for carboniser hot gas casting, (xiii) multiple stirrer unit, (xiv) welding electrode holder, (xv) spiral type dust collector, (xvi) mild steel condenser receiver unit, (xvii) instrument for pressure test on paint panels, (xviii) spray booths, 2 nos, (xix) auto speed control device, (xx) auto self-starter relay, (xxi) stainless steel condenser (xxii) tar distallation plant, (xxiii) reduction drive for vacuum pump, polarimeter tubes, 10 sets, (xxiv) drive for the Lang Emulsifier unit, (xxv) stainless steel water tank, (xxvi) stainless steel desiccator, (xxvii) model of the Photo - chlorination plant, (xxviii) model of the paper lifting vat, (xxix) laboratory equipment such as funnel stands, burette stands, wooden equipment like switch boards, reapers, patterns for castings, etc.

SERVICES

Introduction: The installation and upkeep of services such as electricity, gas, steam and water, which are the lifelines for the functioning of the Laboratory are amongst the functions of the General Engineering Division. Steps taken for maintenance of the services are outlined below.

Work during the year :

1. Electricity:

(i) Two 200 Amp. O. C. Bs., one each for the workshop and the furnace room, were erected and commissioned. The other existing O. C. Bs. were serviced.

(ii) A T-joint made in the L. T. C. plant supply cable and connection given to the tar distillation plant.

(iii) Electrical installations in the L. T. C. laboratory and the Post Office were completed.

(iv) Three - phase supply was extended to the L. T. C. laboratory and single - phase supply to the Post Office and the gate lodge.

(v) Electrical wiring in the Instrumentation Section and rewiring in the Fats and Oils Division were done.

2. Gas:

Separate gas supply mains were provided to the L.T.C. laboratory, and modifications carried in the main gas supply lines of the Laboratory.

Repairs have been made in the gas tanks which had rusted and were developing leakages. As the gas consumption in the Laboratory has nearly doubled, the re-conditioning of the gas tanks is felt necessary.

3. Steam:

(i) A 2" main steam line was extended from pilot plant No. 2 to pilot plant No. 4.

(ii) Foundations were laid for the Werhle Werke and Cochrane boilers at the Fat Splitting and Fatty Acid Distillation pilot plant site. Erection of the Werhle Werke boiler is in progress.

(iii) Steam grid has been finalised. Provisional arrangement has been made for supplying steam to the tar distillation plant by installing a small vertical boiler.

4. Water :

(i) Two tube wells were completed, and the submersible pumps were put into operation. The second tube well drilled to a depth of 30 ft. was not giving adequate supply. Its supply will be tested again during the next monsoon.

(ii) Boring of a third tube well was commenced near the site for the staff quarters. The work is in progress.

(iii) All the black pipe water lines inside the main building have been replaced with G. I. pipes.

(iv) Separate lines were provided in the Laboratory and the pilot plant buildings for the unfiltered, filtered and softened water.

(v) The pump house on the well acquired on lease was submerged in water during the rainy season. Necessary repairs were done to the pump sets.

5. Repairs and maintenance :

Necessary repairs have been done to the following :

(i) Gland and stirrer assembly of the vat fermenter of the Biochemistry Division, (ii) lifting jack, (iii) paper cutting machine, (iv) rag cutter, (v) paper press, (vi) gas tanks, 2 Nos., of the gas house, (vii) ice plant brine pump motor, (viii) electrode boiler, (ix) distilled water still, (x) surface grinder, (xi) electric motors, (xii) vaccum pump of the Fats and Oils Division, (xiii) hand milling machine, (xiv)

rotary digester, (xv) pulp beater of the hand-made paper and cellulose Section, (xvi) ice plant, refrigerators, water coolers, air-conditioners, automobiles, etc.

The low-temperature carbonisation pilot plant was kept in operation and regular maintenance work (both mechanical and electrical) was attended to.

6. Civil and Sanitary works: (See **Buildings** under GENERAL ADMINISTRATION).

Library

A. Books

Books in stock on April 1, 1958	...	7748
Books taken in stock during the year	...	1693
Total number of books in the library on March 31. 1959	...	9441

B. Periodicals

Journals subscribed as on April 1, 1958	...	381
Journals discontinued during the year	...	3
New journals subscribed for during the year	...	48
Total number of journals subscribed for on March 31, 1959	...	426
Total number of bound volumes in stock (approx.)		6500

C. Deletions and additions

Ceased publication:

1. A. I. R. Selections (India).
2. Fuel Abstracts (U. K.).
3. Photorama (Belgium).

Additions:

1. American Documentation, New York (USA).
2. American Institute of Chemical Engineers Journal, Virginia (USA).
3. Biological Reviews, Cambridge (UK).
4. British Medical Journal, London (UK).
5. British Journal of Pharmacology and Chemotherapy. London (UK).
6. Biochemical Pharmacology, London (UK).
7. Canadian Journal of Microbiology, Ottawa (Canada).
8. Canadian Journal of Physics, Ottawa (Canada).
9. Cancer Research, Chicago (USA).
10. Current Engineering Practice, Bombay (India).
11. Chemical Engineering Group : Proceedings of the Society of the Chemical Industry, London (UK).
12. Chemist Analyst, New Jersey (USA).
13. Colourage, Bombay (India).
14. Experimental Cell Research, New York (USA).
15 Ergonomics, London (UK).
16. Federation Proceedings, Washington (USA).
17. Finish, Bombay (India).
18. Grinding and Polishing, Den Bosch (Netherlands).
19. IMDA Bulletin, Bombay (India).
20. International Abstracts of Biological Sciences, London (UK).

21. Journal of the American Water Works Association, New York (USA).

22. Journal of Applied Physics, New York (USA).

23. Journal of Biophysical and Biochemical Cytology, Baltimore (USA).

24. Journal of Histochemistry and Cytochemistry, Baltimore (USA).

25. Journal of Biochemistry (Japan).

26. Journal of Biological Chemistry, Baltimore (USA).

27. Journal of Optical Society of America, Lancaster (USA).

28. Library Association Record, London (UK).

29. Micro Chemica Acta, Vienna (Germany).

30. Monthly Statistics of the Foreign Trade of India, New Delhi (India).

31. NML Technical Journal, Jamshedpur (India).

32. Nutrition Reviews New York (USA).

33. Nutrition Abstracts and Reviews, Aberdeenshire (UK).

34. Office Management, New York (USA).

35 Oil Mill Gazetteer, Texas (USA).

36. Paper Salesman, Bombay (India).

37. Paint Technology, Middlesex (UK).

38. Physiological Reviews, Washington (USA).

39. Popular Science, New York (USA).

40. Popular Plastics, Bombay (India).

41. Quarterly Review of Biology, Baltimore (USA)

42. Research Management, New York (USA).

43. Silk and Rayon Industries of India, Bombay (India).

44. Timber Dryers' and Preservers' Association of India Journal, Dehra Dun (India).

45. Veterinary Bulletin, England (UK).

46. Virology, New York (USA).

D. Changed titles

	Old title	*New title*
1.	American Perfumer and Essential Oil Review, New York (USA).	American Perfumer and Aromatics, New York (USA).
2.	Art and Industry, London (UK).	Design for Industry, London (UK).
3.	Colonial Geology and Mineral Resources, London (UK).	Overseas Geology and Mineral Resources, London (UK).
4.	Journal of the Scientific Research Institute, Tokyo (Japan).	Scientific Papers of the Institute of Physics and Chemical Research, Tokyo (Japan).
5.	Official Air Line Guide (India), New Delhi (India).	Indian Air Lines Corporation (India), New Delhi (India).
6.	Translated Contents List of Russian Periodicals, London (UK).	L. L. U. Translation Bulletin, London (UK).

E. Journals received gratis

	Title	*Courtesy of*
1.	China Today	Embassy of the Peoples Republic of China in India, New Delhi.
2.	China Pictorial	

3.	Democratic Republic of Viet-Nam	Consulate - General of the Democratic Republic of Viet-Nam in India, New Delhi.
4.	Korea Today	Embassy of the Democratic People's Republic of Korea in India, New Delhi.
5.	NML Technical Journal	National Metallurgical Laboratory, Jamshedpur.
6.	Silk and Rayon Industries of India	Secretary, Silk and Art Silk Mill's Research Association, Bombay.
7.	Soviet Land	Information Department of the USSR Embassy in India, Bombay.
8.	Tropical Abstracts	Royal Netherlands Embassy, New Delhi.

F. Exchange

Annual reports from the following institutions were received.

1. Sindri Fertilizers and Chemicals (Private) Ltd., Bihar.
2. Central Drug Research Institute, Lucknow.
3. Railway Testing and Research Centre, Lucknow.
4. Nutrition Research Laboratories, Coonoor.
5. Sugarcane Breeding Institute, Coimbatore.
6. Council of Scientific & Industrial Research, New Delhi.
7. U. S. Government Printing Office, Washington.
8. Indian Central Jute Committee, Calcutta.
9. Central Leather Research Institute, Madras.
10. British Gelatine and Glue Research Association, London.

11. National Metallurgical Laboratory, Jamshedpur.

12. Haffkine Institute, Bombay.

13. Tata Chemicals, Bombay.

G. General

Readers from outside permitted to consult the library. 62

Reprints and publications (other than Annual Report) sent to research institutes and industries on request. 50

Copies of Annual Report 1957-58 sent to research and educational institutions and laboratories. 459

Preprinted copies of symposium on 'Cottonseed and its by-products' sent to participants and institutes. 240

Membership

Members of the staff borrowing books and journals from the library as on April 1, 1958. 113

New members. 99

Members borrowing books and journals as on April 1, 1959. 212

Backsets

Backsets of the following journals have been purchased.

1. Arkiv fur Mikrobiologie, Berlin (Germany). Vols. 14 - 31

2. Journal of Scientific Instruments, London (UK). ,, 1 - 19

3. Review of Scientific Instruments, New York (USA). ,, 1 - 3

4. Journal of American Chemical Society, Easton (USA). ,, 1 - 22

5. Helvetica Chimica Acta, Basel (Switzerland). ,, 1 - 14

6.	Berichte der Deutschen Keramischen Gesellschaft Weinheim. (Germany).	,,	1 - 7 ,, 17 - 20
7.	Journal of the American Ceramic Society, Easton (USA).	,,	1 - 19
8.	American Ceramic Abstracts, Easton (USA).	,,	1 - 35
9.	American Institute of Chemical Engineers Journal, Virginia (USA).	,,	1 - 3
10.	Transactions of the Institute of Chemical Engineers, London (UK).	,,	1 - 19
11.	Chemical Abstracts, Ohio (USA).	,,	4 - 5
12.	Chemical Reviews, Baltimore (USA).	,,	1 - 37

COLLOQUIUM

Secretary : Dr. M. B. Naidu.

1958

Speaker	*Subject*	*Date*
Shri T. Satyagopal, Coal Survey Station, (Central Fuel Research Institute), Hyderabad.	Use of cocotheline as an analytical reagent.	May 3
Prof. S.K.K. Jatkar, Professor of Chemistry, Poona University, Poona.	Strong electrolytes.	May 9
Shri Y. V. Subba Rao, Regional Research Laboratory, Hyderabad.	Extraction and recovery of tar acids from ammoniacal liquors of low-temperature carbonisation plants.	June 21
Dr. V. S. Subrahmanyam, Regional Research Labroratory, Hyderabad.	Studies on complex thiocyanates of cobalt and nickel.	July 11

Dr. Y. Nayudamma, Director, Central Leather Research Institute, Madras.	Some aspects of leather science.	July 30
Dr. P. B. Sattur, Regional Research Laboratory, Hyderabad.	Studies on phenylethylamine and its derivatives.	August 23
Rev. Dr. L. M. Yeddanapalli, Professor of Chemistry, Layola College, Madras.	Kinetics and mechanism of formation of phenol-formaldehyde resins.	August 25
Dr. M. D. Narasimhan, Regional Research Laboratory, Hyderabad.	Mechanism of sintering in the presence of a liquid phase.	August 30
Dr. A. P. Mahadevan, M/s. Hindustan Lever Ltd., Bombay.	Synthesis of some unsaturated fatty acids.	September 5
Shri A. Rahman, Assistant Director, Central Building Research Institute, Roorkee.	Selection and opportunism in science.	September 8
Dr. Claus Franzke, Assistant Professor, Institute for Food Chemistry and Technology, Humboldt University, (East Germany).	Preparation of normal oleic, linoleic acids using adsorption chromatography.	December 10
Dr. B.S.R. Sastry, Regional Research Laboratory, Hyderabad.	Liquid immiscibility in the system lithia-boric oxide-silica.	December 31

1959

Lt. Gen. Sir Harold Williams, Director, Central Building Research Institute, Roorkee.	Research in India and its approach.	January 9

Dr. Ivan Malek, Director, Biological Institute of Czechoslovakian Academy of Sciences, Prague, (Czechoslovakia).	Continuous cultivation of micro-organisms.	February 3
Dr. Otto Isler, Head of Department of Organic Chemistry, Hoffmann La Roche, Switzerland.	Synthesis of fat-soluble vitamins with special reference to vitamin A.	March 6

DISTINGUISHED VISITORS

1958

Name and address	*Date*
Dr. K. Mori, Instructor, Horticultural College, Chiha University, Japan.	April 18
Herr Hermann Soergel, Commercial Adviser, Trade Representation of the German Democratic Republic in India, Bombay.	April 19 & December 26
Herr Kurt Fabian, Deputy Trade Representative, Trade Representation of the German Democratic Republic in India, Bombay.	April 19
Dr. P. De. Jong, Technical Manager, Tata Fison Private Ltd., Bangalore.	April 23
Shri S. Masuda, Director and General Manager of India Office, Kinoshita & Co, Ltd., New Delhi.	May 5
Dr. Phil Hoerft Krueger, Cultural Adviser to the Trade Representation of the German Democratic Republic in India, New Delhi.	May 5
Hon'ble Mr. Kamalluddin Mohamed, Minister for Agriculture, Lands and Fisheries, Trinidad.	July 24
Hon'ble Mr. D. C. Granado, Minister for Labour, Co-operative Development and Social Services, Trinidad.	July 24

Dr. Y. Nayudamma, Director, Central Leather Research Institute, Madras.	July 29
Kumari M. Masani, Director, External Services, All-India Radio, Hyderabad.	July 17
Shri C. J. S. Darosse, Manager, Imperial Chemical Industries, Ahmedabad.	July 17
Prof. Humayun Kabir, Minister for Scientific Research and Cultural Affairs, Government of India, New Delhi.	August 8
Shri D. S. Reddy, Vice-Chancellor, Osmania University, Hyderabad.	August 18
Dr. Manomohan Das, Dy. Minister for Scientific Research and Cultural Affairs, Government of India, New Delhi.	September 5
Shri N. Sanjiva Reddy, Chief Minister, Andhra Pradesh.	September 13
Lala Charat Ram, Managing Director, The D. C. M. Chemical works New Delhi.	September 13
Dr. S. Bhagawantam, Director, Indian Institute of Science, Bangalore.	September 13
Shri Anil De, Director, of Industries Andhra Pradesh.	September 13
Shri Syed Kazim, Director, Mines and Geology, Andhra Pradesh.	September 13
Mr. A. G. Natradze Mr. V. Dsoniakovshki Mr. A. Hskov. — Pharmaceuticals and Drugs Experts Delegation from Russia.	October 3
Shri Dinabandhu Sahu, Minister, Industries and Mining, Bhubhaneshwer, Orissa.	October 24
Dr. Claus Franzke, Asst. Professor, Humboldt University, (E. Germany.)	December 11

Mr. S. W. Turner, Technical Manager, Hindustan Lever Ltd., Bombay.	December 13
Dr. Hager, Director, Lurgi Gesellschaft fur Warmetechnik W. Germany.	December 13 February 17 (1959)
Dr. M. Habibullah, Director of Industries, Jammu and Kashmir Government.	December 17

1959

Lt. Gen. Sir H. Williams, Director, Central Building Research Institute, Roorkee.	January 6
Mr. Kurt Bransch, Berlin.	January 28
Academician Dr. I. Malek, Director, Biological Institute of Czechoslovakian, Academy of Sciences, Prague.	February 3
Dr. B. Shariat, Journalist, Tehran, Iran.	February 9
Mr. Jean Lequiller, Cultural Counsellor, French Embasy, New Delhi.	February 10
Shri C. P. Shah, Technical Expert, Bombay Potteries and Tiles Ltd., Bombay.	February 12
Mr. George Senf, Representative, Carl Zeiss, Jena, Germany.	February 16
Mr. Helmut Maier, Representative, Karl Kolb, Germany.	February 16
Dr. H. H. Koppers, Director, Heinrich Koppers Ltd., Germany.	February 16
His Highness Shri Yadvendra Singh, Maharaja of Patiala.	February 18
Dr. Ing. Tito Borelli, Vice-Director of Monticatini, Milan, Italy.	March 11
Dr. Ing. P. Gadina, Engineer, Monticatini, Milan, Italy.	March 11
Mr. W. G. Brown, Dy. Chief and Minister Counseller, U. S. Embassy, New Delhi, accompanied by Mrs. Brown.	March 11

APPENDICES

1. PERSONNEL OF THE EXECUTIVE COUNCIL, REGIONAL RESEARCH LABORATORY, HYDERABAD, DURING 1958-59.

Chairman

Shri N. Sanjiva Reddy, Chief Minister, Government of Andhra Pradesh, Hyderabad.

Members

Prof. T. R. Seshadri, Professor of Chemistry, University of Delhi, Delhi - 8.

Dr. S. Bhagawantam, Director, Indian Institute of Science, Bangalore - 3.

Lala Charat Ram, The D. C. M. Chemical work Private Ltd., Delhi - 6.

Dr. G. P. Kane, Senior Industrial Adviser (Chemicals), Ministry of Commerce & Industry, Government of India, New Delhi.

Dr. V. T. Athavale, Senior Research Officer, Atomic Energy Establishment, Trombay, Bombay - 28.

Shri Anil De, Director, of Industries Andhr Pradesh, Hyderabad.

Shri C. V. D. Murthy, Director, Technical Education, Hyderabad.

Shri Syed Kazim, Director, Mines and Geology, Andhra Pradesh, Hyderabad.

Shri D. S. Reddi, Vice-Chancellor, Osmania University, Hyderabad.

Prof. C. Mahadevan, Professor of Geology, Andhra University, Waltair.

Ex-officio Members

Prof. M. S. Thacker, Director - General, Scientific & Industrial Research, New Delhi.

Shri A. V. Venkateswaran, Financial Adviser to C. S. I. R., New Delhi.

Dr. S. Husain Zaheer, Director, Regional Research Laboratory, Hyderabad.

2. PERSONNEL OF THE SCIENTIFIC SUB-COMMITTEE OF THE EXECUTIVE COUNCIL.

Chairman

Dr. S. Husain Zaheer

Members

Prof. T. R. Seshadri

Dr. S. Bhagawantam

Lala Charat Ram

Dr. G. P. Kane

Dr. V. T. Athavale

Shri Anil De

Shri C. V. D. Murthy

Shri Syed Kazim

Prof. C. Mahadevan

Co-opted Members

Shri P. A. Narielwala, Director, Tata Industries (Private) Limited, Bombay - 1.

Shri M. N. Kamlani, Member (Engineering), Railway Board, New Delhi.

Shri C. P. Shah, Technical Expert, Bombay Potteries & Tiles Ltd., Kurla, Bombay.

Dr. Atma Ram, Director, Central Glass & Ceramic Research Institute, Calcutta - 32.

Shri T. M. Jagtiani, Operative Director, Singareni Collieries Co.. Ltd.. Hyderabad.

3. PERSONNEL OF THE FINANCE AND BUILDINGS SUB-COMMITTEE OF THE EXECUTIVE COUNCIL.

Chairman

Prof. M. S. Thacker

Members

Shri A. V. Venkateswaran

Shri Anil De

Dr. S. Husain Zaheer

The Chief Engineer (Building), Andhra Pradesh Government.

4. PERSONNEL OF THE EXPERTS' SUB-COMMITTEE OF THE EXECUTIVE COUNCIL.

Chairman

Dr. S. Husain Zaheer

Shri Anil De

Shri C. V. D. Murthy

Shri Syed Kazim

ERRATA

Page	*Line from top*	*For*	*Read*
viii	21	Waheedududdin	Waheeduddin
19	29	sulphated salts	sulphated salt
21	26	exepeller	expeller
23	35	(on the still charge of	(on the still charge) of
32	8	Wehrlwerke	Werhle Werke
32	34	per lb., and while	per lb., while
33	33	sufficientlv	sufficiently
36	11	to 6 per cent	of 6 per cent
36	16	styrention	styrenation
37	7	*terl*	*tert*
67	7	*acetyl–Co* A	acetyl–Co A
68	14	*acetyl–Co* A	acetyl–Co A
68	16	substitute	substrate
68	19	*acetyl–Co* A	acetyl–Co A
69	15	cane sugar	a readily available sugar
	7	prepaped	prepared
77	29	component	components
81	12	50 ml.	500 ml.
82	27	yieds	yields
84	36	3 : 3 : 31.5,	3 : 3 : 1.5,
85	1	reage nt	reagent